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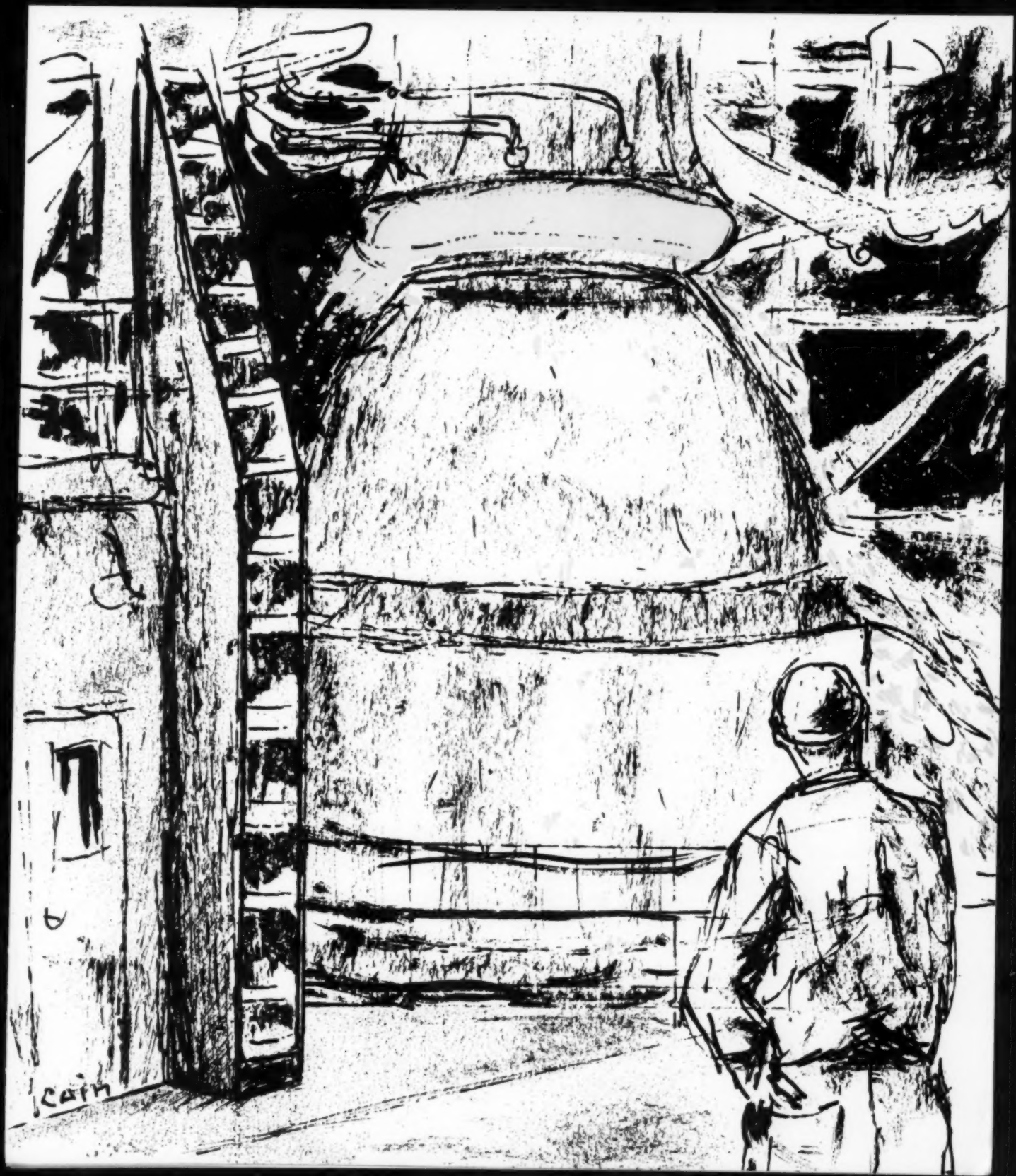

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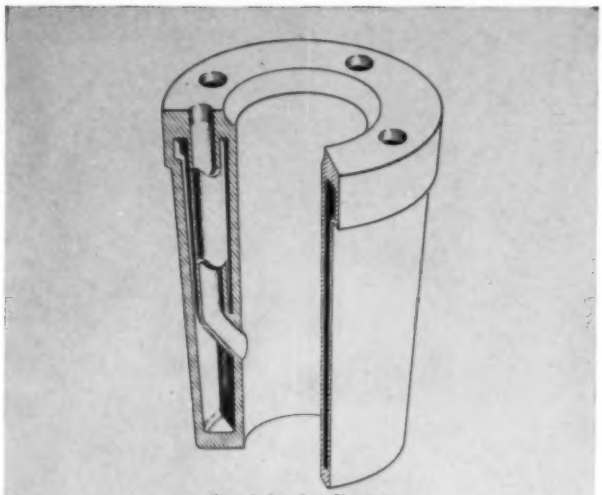

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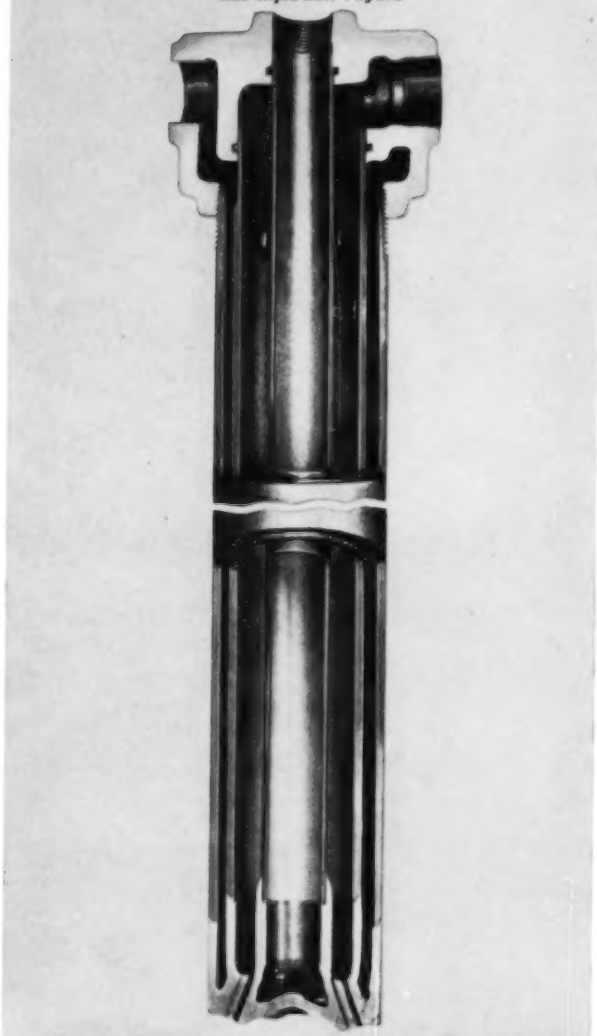

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Vol. 13, No. 9
September, 1961

COVER: Blowing one of CF&I's two basic oxygen furnaces is the subject of this month's cover by David Cain. Articles on America's four new basic oxygen steel plants are found in this issue, together with a detailed description of the kinetics of the processes.

contents 581
engineering consultants 584
personnel 584
books 589
reporter 595
editorial 607
personals 602
education 611
news of your profession 645
advertisers index 652
coming events 652

Process Kinetics of Oxygen Steelmaking 613
by W. O. Philbrook

Basic Oxygen Steelmaking in Colorado 621
by George Grosvenor

Cleveland Receives Basic Oxygen Steelmaking 625
by J. A. Glasgow

Great Lakes' Basic Oxygen Furnaces 628
by R. B. Morgan

Basic Oxygen Furnace Linings at Aliquippa 631
by J. N. Albaugh

Air Pollution Control in Oxygen Steelmaking 632
by J. H. Smith

Sharon Steel's Kaldo Furnaces 635
by R. N. Merk and D. R. Berg

Sweden's Second Kaldo Plant 638
by Carl Sebardt

Electron Beam Symposium 641
by Robert Bakish

What's Happening to Beryllium 598

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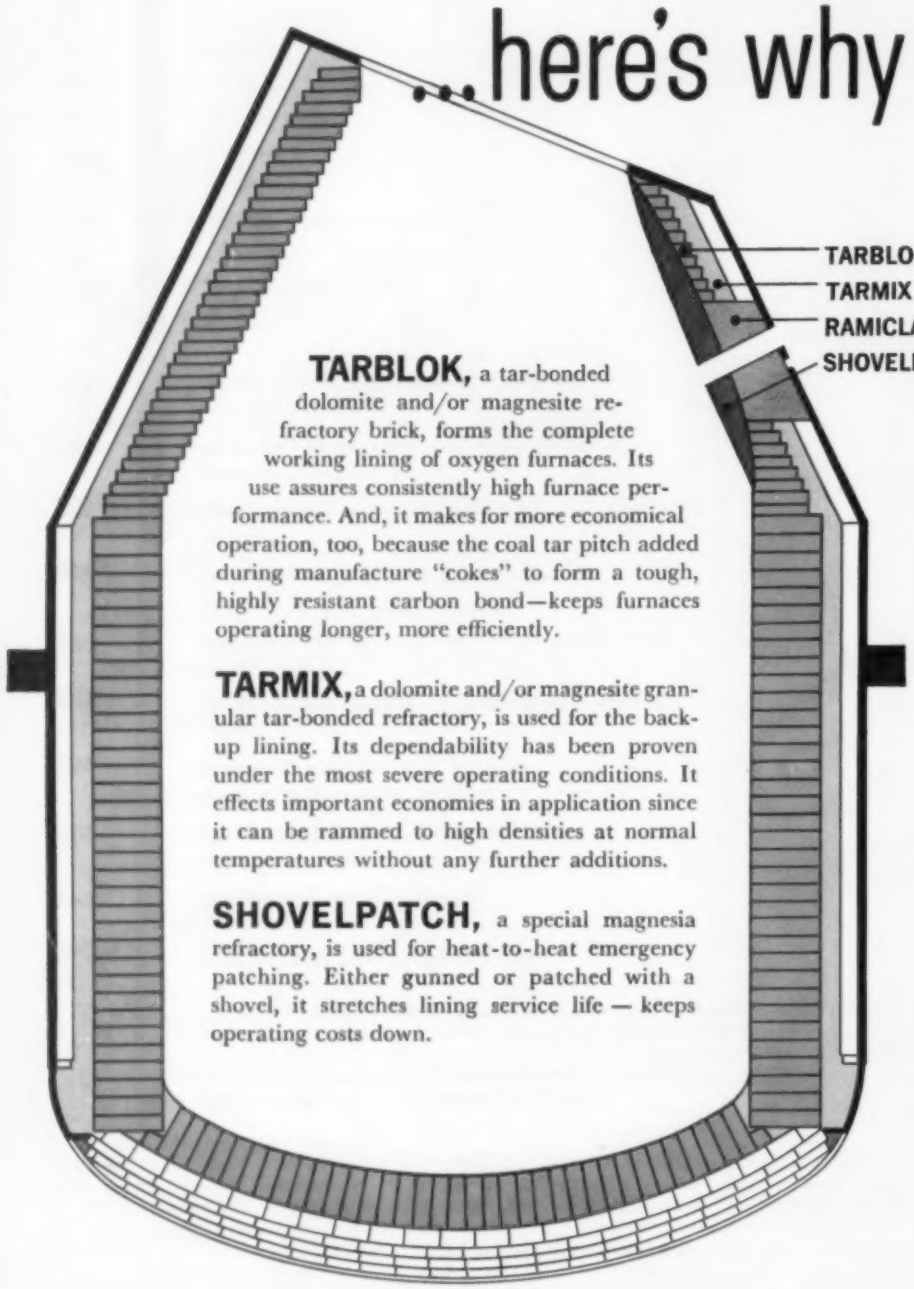

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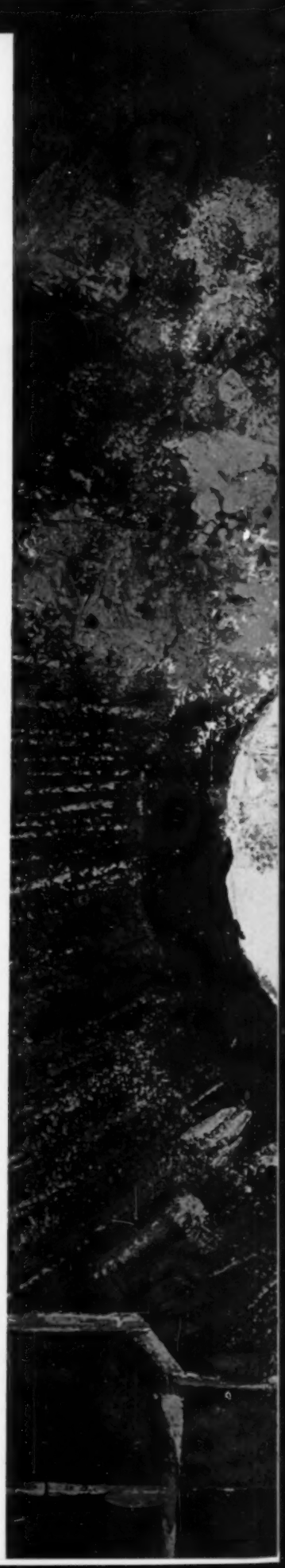

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Research Metallurgist, B.S. Met. Eng., age 42, married, three children. Fifteen years experience aluminum base alloys, free machining and high strength alloy development, investigation of aluminum powder alloys and aluminum fibers, magnesium base alloys for forgings and elevated temperature use, process variables to improve magnesium base alloy; two years metallurgy of uranium and uranium base alloy; two years study oxidation of copper alloys, publications and patents. Available immediately. Prefer West but will relocate. M-260.

Sales-Metal Products: age 27, Physical Metallurgy. Prefer Western United States. Home: Washington. Four years experience as metallurgist in rolling mills, aircraft and electronics, heat transfer and forming problems, quality control. Se-1693.

Metallurgist, B.S. Eng. Physics, age 37, Reg. P.E. Eleven years diversified experience non-profit organization, forging and steel industry, both research and plant operation. Have strong inclination and desire for research opportunity in ferrous, high-temperature, or refractory metal field. Prefer South, Southwest, West. M-2229-Chicago.

Research and Development Staff Metallurgist, Ph.D. '59, B.Sc. Met.E '53, age 32. Four years university research, two years industrial research, one year production engineering experience. Thorough knowledge material science, flow and fracture of metals, phase transformation and phase equilibria, X-ray and electron diffraction, physics of metals. Knowledge solid state for possible integration into materials science in semiconductor industry. Domestic or foreign. M-2230-Chicago.

Concentrator Superintendent, Assistant-Copper: age 36, Met.E., AIME. Prefer Western United States. Home: Arizona. $900. Recently superintendent of 15,000 tpd copper mill, moly plant, 1200 tpd slime leaching plant. Formerly foreman of 200 tpd contact acid plant, foreman of tank house for electrodeposition and electro-refining of copper. Se-192.

Metallurgist, Assistant Superintendent-Ore Processing: age 32, Met.E., AIME. Prefer South America. Home: Colorado. $7500. Over four years testing and control of various phases of process in zinc smelting (horizontal sintering machines, retort smelting, magnetic separation) and processing copper ore (crushing, grinding, floatation, reagent preparation and control) and processing tungsten ore (crushing, grinding, tabline). Se-140.

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Consultant: age 70, Met.E., AIME. Prefer Southwest United States. Home: Nevada. $400. Twenty years experience in private commercial production of nonferrous hi-purity ingot metals in the United States and foreign countries. Twenty-five years research and development extractive process metallurgy. Se-1241.

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(Continued on page 608)

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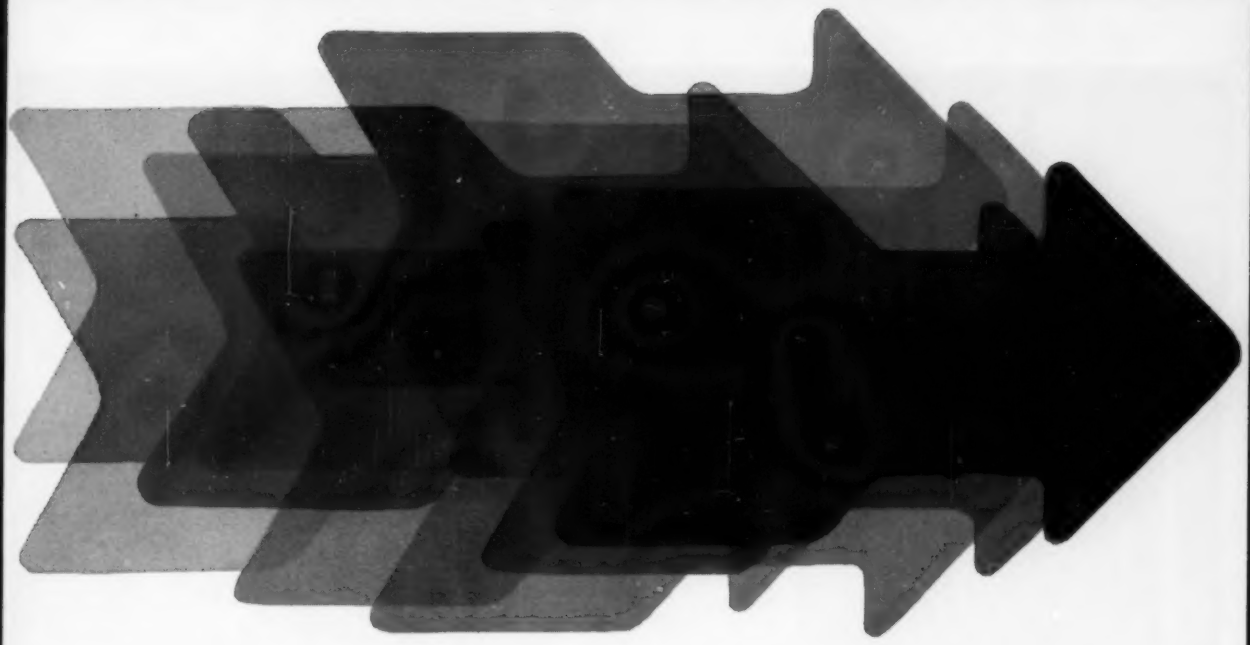

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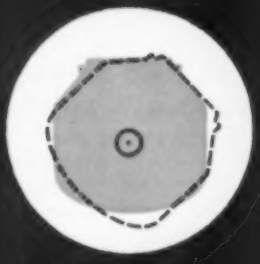

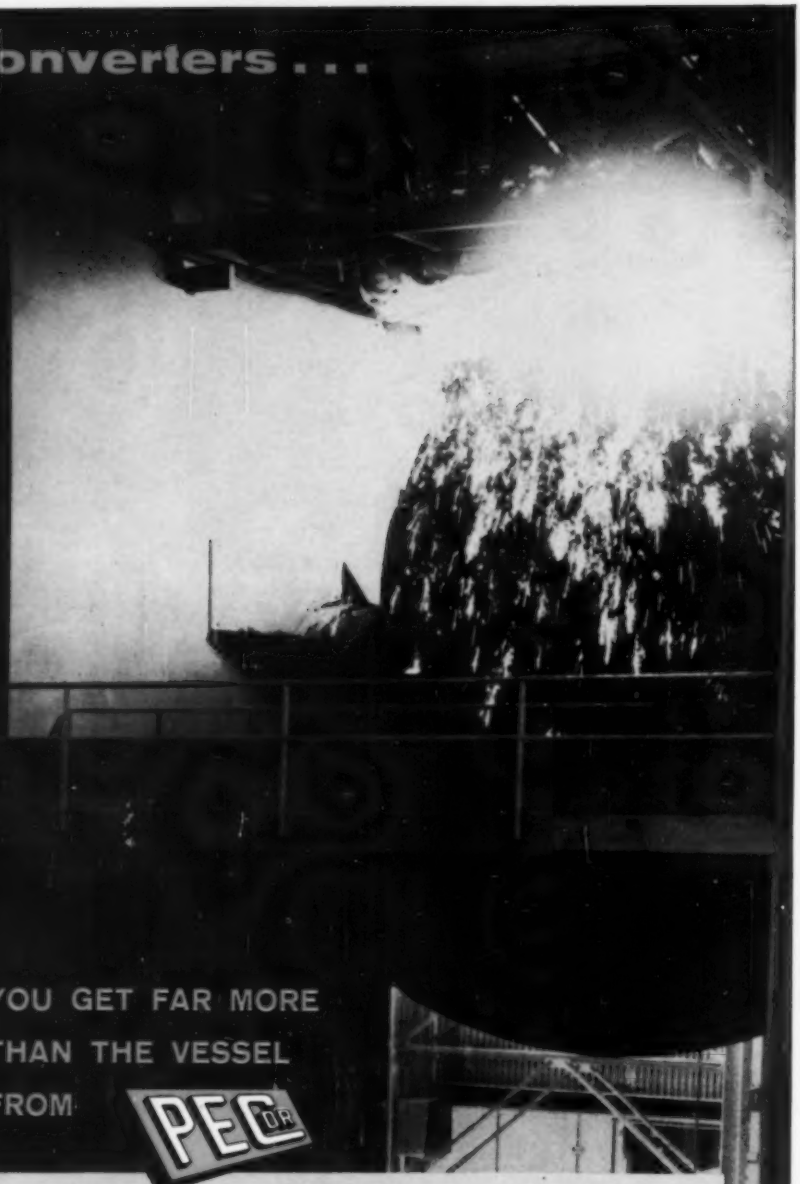

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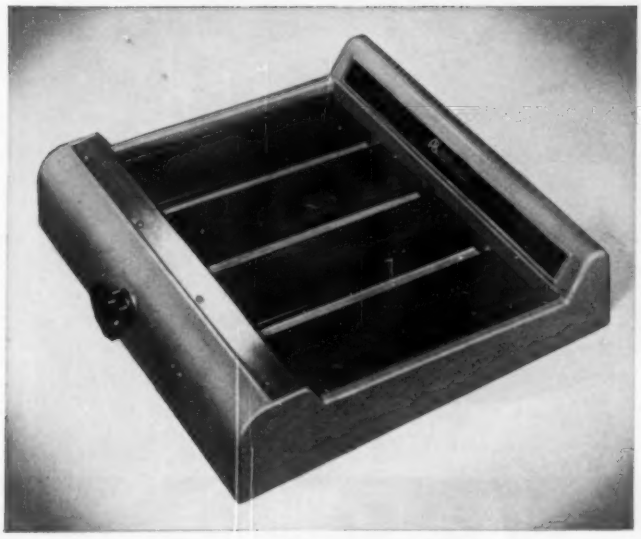

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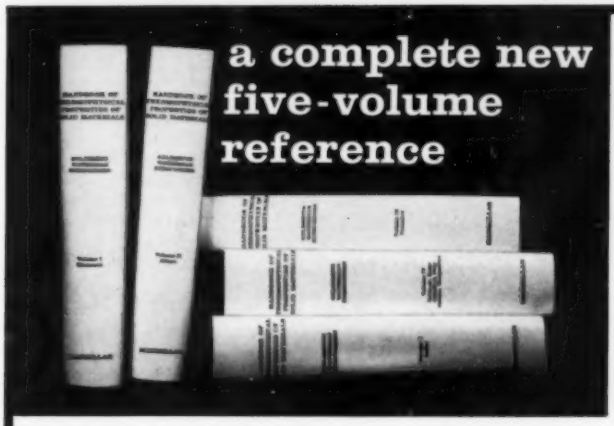

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Thermodynamics

Thermodynamics by G. N. Lewis and M. Randall, Second Edition, Revised by K. S. Pitzer and L. Brewer, McGraw-Hill Book Co., Inc., 723 pp., $12.50, 1961. *Reviewed by John F. Elliott, Massachusetts Institute of Technology.* •

Many readers who were brought up in the Lewis and Randall tradition will find reading this text to be a sentimental journey into the past, and new students of the subject will now be able to share this tradition in a modern setting. Professors Pitzer and Brewer are to be congratulated on retaining the clear and lucid style of the original. This has been done successfully even in those chapters covering topics which were not in the original.

Even with the addition of a number of chapters dealing with phenomena, including irreversible processes, which were not covered in the original, the text essentially deals with chemical thermodynamics. Students and workers in the fields of metals and materials will find much of specific value to them. It is encouraging to note that the metallurgist's contributions to the knowledge of the thermodynamics of metallic solutions and to high temperature inorganic chemistry have been recognized by the chemists.

Preservation of the Lewis and Randall tradition apparently has forced the present authors to retain the view of the 1920's of the basic Laws of Thermodynamics. There is no reflection of the exciting discussions currently in progress concerning the logical development and the meaning of these Laws. The circuitous logic of explaining heat in terms of temperature and the reverse is retained. Also, in essence the Second Law is called upon and used, i.e. heat flows from a higher temperature to a lower temperature prior to its formal development.

The symbolism is essentially Lewis' and Randall's. Consequently, some of the helpful usages of Wagner have not been adopted. In fact, his interaction coefficient ϵ is converted to a k. Another change which may be somewhat confusing is that the work term in the First Law is positive instead of negative as is usually the case.

On the whole this is a valuable and useful text for chemists and metallurgists. It has a very broad coverage of the subject and will undoubtedly take its place on many shelves beside the original.

Extractive Metallurgy

Extractive Metallurgy of Copper, Nickel, and Cobalt, edited by Paul Queneau, Interscience Publishers, Inc., 633 pp., $22.50 (AIME members price $18.00), 1961. *Reviewed by A. W. Schlechten, University of Missouri.* •

This volume is based on the International Symposium held in New York in February 1960 and sponsored by the Extractive Metallurgy Division of The Metallurgical Society. It is the first volume in a series planned by the EMD to describe the present status and recent developments in the important areas of nonferrous extractive metallurgy. The present volume and the ones to follow should be most valuable references for operating metallurgists, researchers, students, and faculty who wish to know present practice in the field, because this assembled information is certainly not available from any other source.

One cannot help but look back and compare this volume with Volume 106 of the TRANSACTIONS which reviewed copper metallurgy as it was 28 years ago. Many subjects discussed in the older book are still with us, but there are also many new subjects such as fluid bed roasting, ion exchange, pressure leaching, solvent extraction, and dialysis that show how far we have come in the intervening years.

Some US copper metallurgists may think the new volume is a little heavy on nickel and cobalt, but all aspects of extractive copper metallurgy are covered, with the possible exception of fire refining. It is unfortunate that the recent paper on poling with natural gas at Phelps Dodge is not included in this collection.

In reading the papers on nickel and cobalt, one is impressed with the variety of processes that are commercially feasible for these two metals, especially in comparison with the practically standardized copper flowsheets. The elaborate processes that are described for some of the nickel and cobalt plants are a tribute both to the inventive genius of the originators and the engineering ability of the operators who make them work.

One of the most valuable papers in the volume is a bibliography on the extractive metallurgy of copper, nickel, and cobalt from 1946 to 1960. This list can be read through to see if one has missed any important papers in the last 15 years, or it may be used as a basis for a study program to become familiar with any aspect of the extractive metallurgy of these metals.

The old TRANSACTIONS volumes on copper, lead, and zinc, and milling methods are collector's items; we predict that this volume will have an equal demand.

* * *

Two bibliographies of interest to metallurgists have recently been published by the Metallwerk Plansee Aktiengesellschaft. They are:

Powder Metallurgy in Nuclear Engineering (1956-1960), by Henry H. Hausner and Helen C. Friedemann, 119 pp., 1961. This bibliography consists of 643 selected references from the literature from 1956 to 1960, most of them annotated and dealing with the application of powder metallurgy methods in nuclear engineering. The references are arranged alphabetically according to authors; there is a sizable subject index at the end of the booklet.

The references in this bibliography were taken from magazine and book literature published in the US, UK, Canada, France, Sweden, Germany, Austria, and also the USSR, and Czechoslovakia.

Uranium Carbides (1956-1960), by Henry H. Hausner and Helen C. Friedmann, 36 pp., 1961. This bibliography lists 209 articles on uranium carbide with short abstracts of most of them.

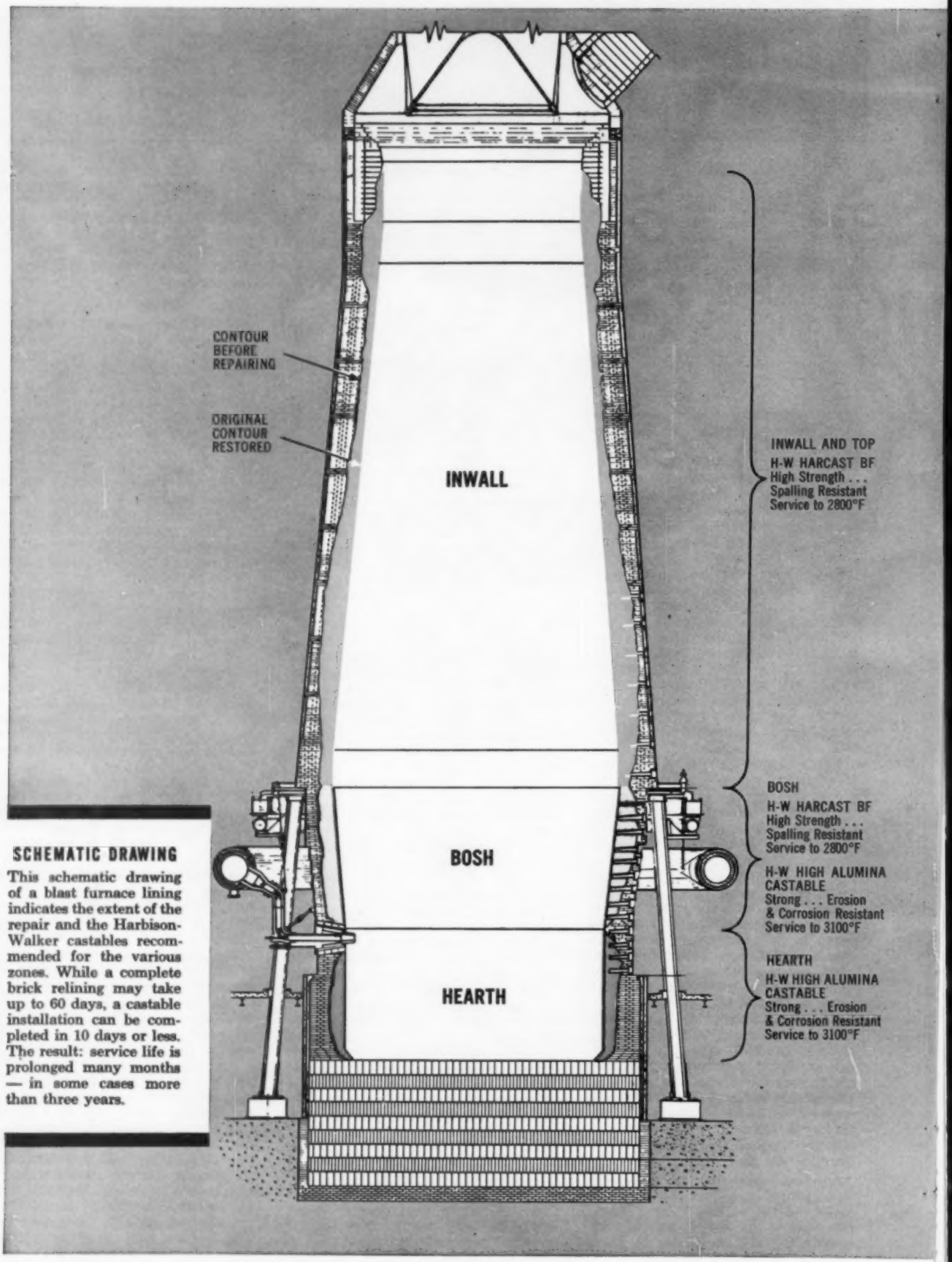
Copies of the two bibliographies are available free of charge from Metallwerk Plansee Aktiengesellschaft, Reutte/Tyrol, Austria.

* * *

Temperature Measurement in Engineering, vol. 2, by H. Dean Baker, E. A. Ryder, and N. H. Baker; John Wiley & Sons, Inc., 510 pp., $13.00, 1961. •

The purpose of the book is to present how-to-do-it directions for the measurement of temperatures in situations which arise in the laboratory, shop, and field. The book is arranged in classes of circumstances where temperature measurements are desired. The techniques employed are complex and require organized explanations. In view of this, the first part is devoted to a discussion and systematic development of the basic methods, resistance-thermometry, and radiation-pyrometry techniques, on an engineering-design-technique basis. Special techniques are described in terms of the greatest adaptability they may possess, but mainly in conjunction with

(Continued on page 608)



SCHEMATIC DRAWING

This schematic drawing of a blast furnace lining indicates the extent of the repair and the Harbison-Walker castables recommended for the various zones. While a complete brick relining may take up to 60 days, a castable installation can be completed in 10 days or less. The result: service life is prolonged many months — in some cases more than three years.

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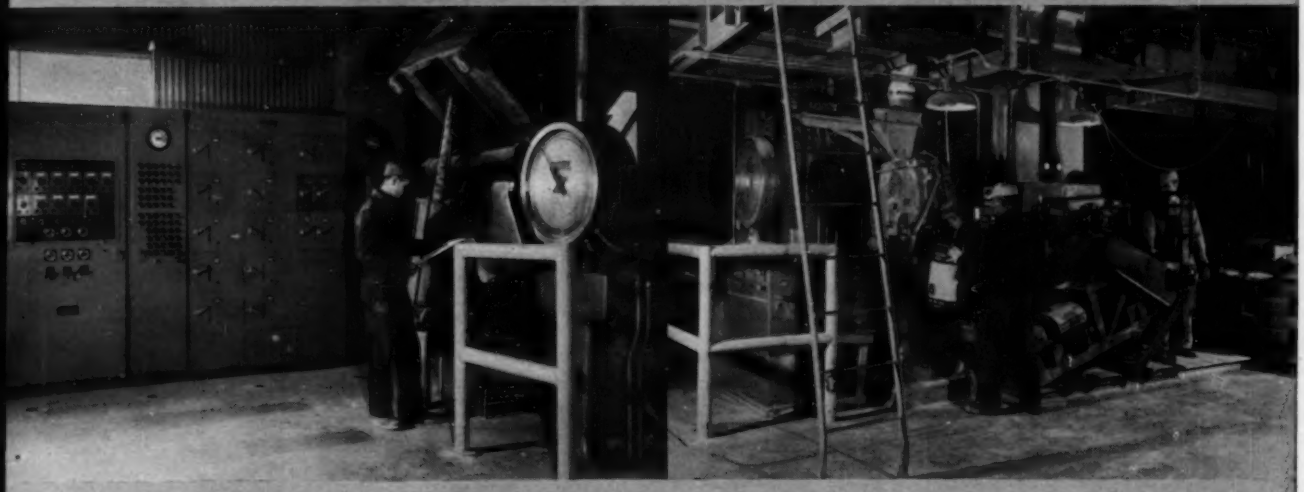

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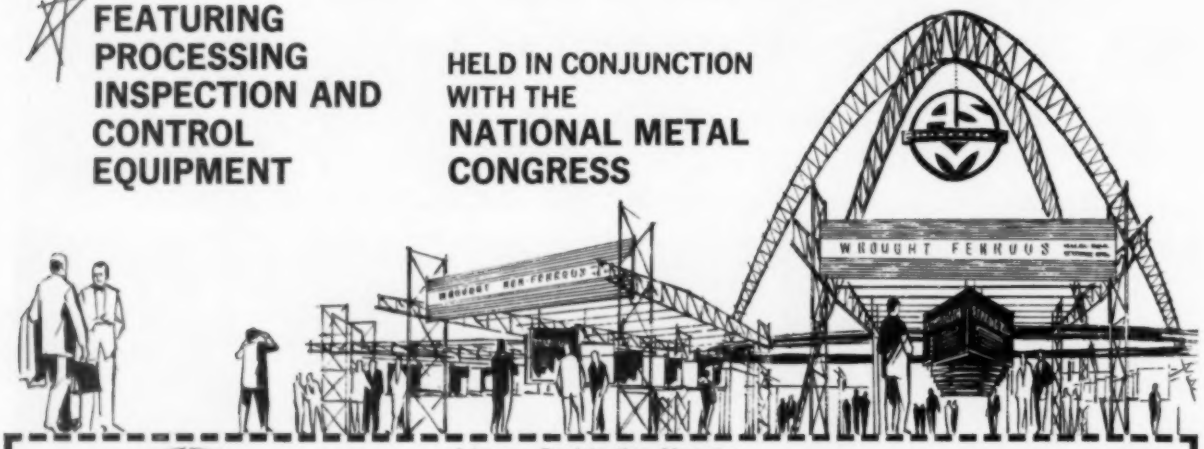

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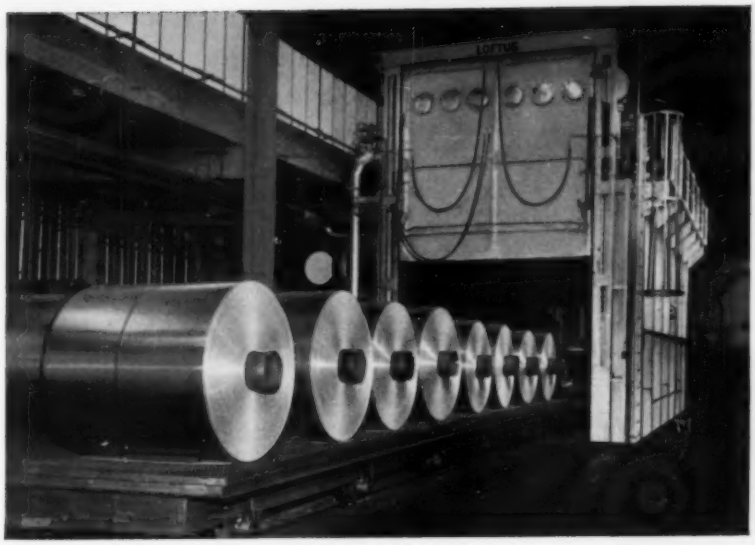

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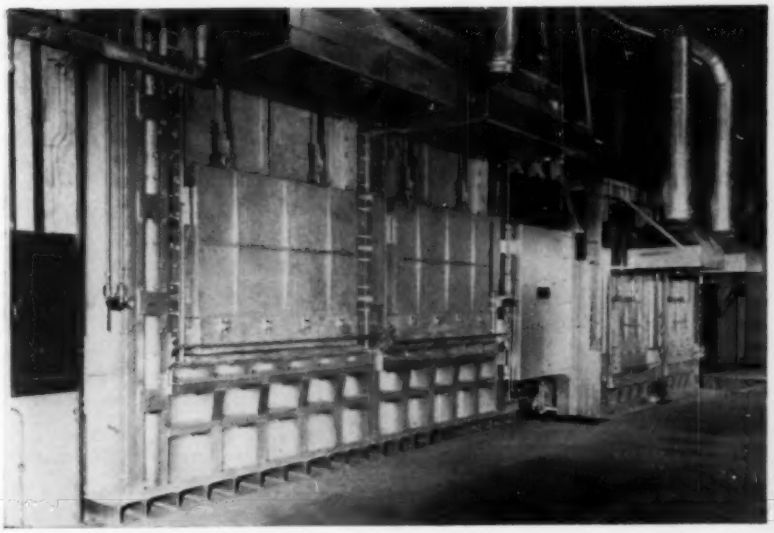

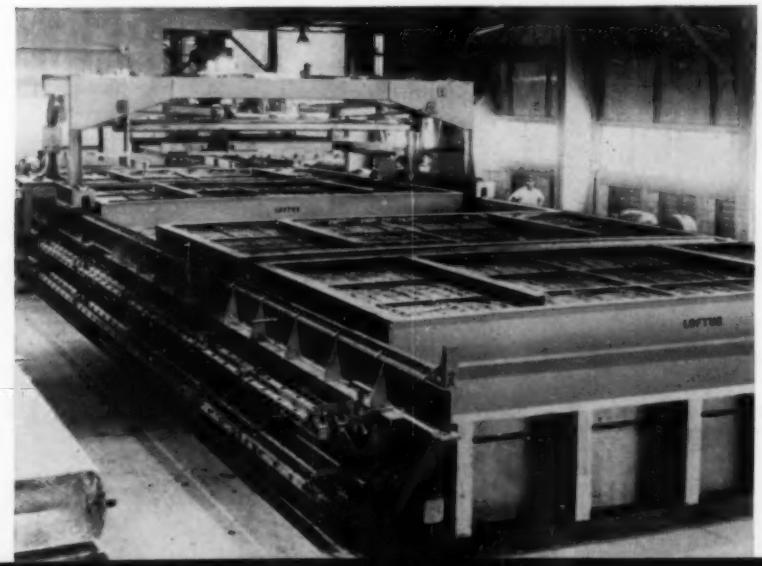

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10
Research-Cottrell precipitators for basic oxygen steelmaking

At present Research-Cottrell has designed a total of ten precipitators for basic oxygen steelmaking with a total capacity of approximately 2,000,000 cubic feet per minute.

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The Cottrell precipitators at the right of the photo remove dust and fume from waste gases at the Kaiser Fontana Plant with 99.8% efficiency. This huge new basic oxygen steel plant was designed and constructed by Kaiser Engineers.

The three Cottrells were designed and built by Research-Cottrell to clean 607,500 cubic feet of gas per minute from the oxygen furnaces. They are installed between the furnace exhaust ducts and the fans serving the three stacks seen at right below.

The high gas cleaning efficiency of this installation is obtained by means of tested and proven Cottrell design features including electrical sectionalization, Opzel collecting plates, and continuous, automatic operation of high tension rappers and M.I. plate rappers.

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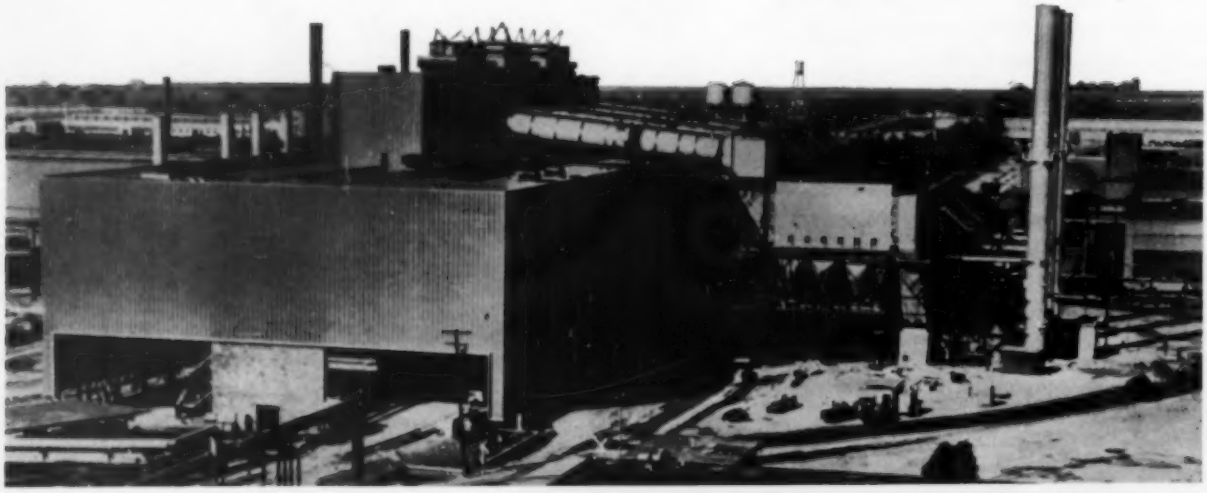

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More Kaldo

Stora Kopparbergs Bergslags A/B is planning to raise its crude steel capacity to 1 million tons per year by replacing its basic bessemer plant at Domnarfvet, Sweden with two, 70-ton, Kaldo converters. The installation is expected to be completed by the Spring 1964, and the converters will be provided with exchange vessels. The Kaldo process is also being introduced to the US, see p. 635 of this issue of JOURNAL OF METALS.

Cb-Zr superconductive alloys

Wah Chang Corp., Albany, Ore. recently reported success in fabricating zirconium alloys containing 25 and 33 pct columbium to sizes less than 0.010 in. in diam and in lengths in excess of 1000 ft. Tests are being conducted on the wire at several laboratories, and preliminary data indicate that the properties are even better than originally observed for the alloys. Test data report critical fields up to 80 k-gauss at current densities in excess of 10^4 amp per sq cm, and it is expected that the wire will be used in electromagnets capable of operating in the 40 to 80 k-gauss range before the end of this year.

Oremet developments

Oregon Metallurgical Corp. recently reported that it has produced molybdenum of exceptionally high purity which is ductile in the as-cast condition, and that it has fabricated tungsten-alloy strip from centrifugal castings.

The ductile molybdenum was produced for the Bureau of Naval Weapons in a pilot plant by reducing molybdenum sulfide to molybdenum with tin. In the process, commercial molybdenite is purified by acid leaching and then briquetted with tin and heated under a hydrogen or argon atmosphere to produce high-purity molybdenum. A 100-lb vacuum-arc-cast ingot produced from the molybdenum analyzed as follows: less than 20 ppm C, O_2, and N_2, and less than 50 ppm S. Before mechanical working, the ingot had the following properties: 37,000 psi yield strength, 46,000 psi ultimate tensile strength, 3.5 pct elongation in 4 diameters, and 3.2 pct reduction in area at room temperature. This is believed to be the first time that molybdenum has been produced pure enough to be ductile in the as-cast condition.

Oremet also announced that is has begun experimental fabrication of tungsten-alloy strip from centrifugally-cast material. Reportedly, the cast material is directly rollable to sheet or strip without prior forging or extrusion, and the rolling operation can be performed at temperatures much lower than expected. For example, a 98 pct W-2 pct Mo alloy was rolled at 1000°C from an as-cast thickness of 0.64 in. to a finished thickness of 0.035 in. without the benefit of an intermediate anneal or recrystallization.

Brazilian oxygen facility

Air Products, Inc. recently announced the start-up of a new oxygen producing facility for the Companhia Siderurgicca Nacional (Brazilian National Steel Co.) at the company's Volta Redonda works, 60 miles northwest of Rio de Janeiro. The 25-million-cu-ft-per-month plant is one of the first plants to supply oxygen for open-hearth steel-making in South America. Built for continuous operation, the plant features a liquid oxygen storage system for 115 metric tons, plus an 8 metric ton gas storage tank, for a four-day back-up supply. In addition, Air Products designed and built the eight oxygen control systems to regulate the introduction of the gas into the open-hearth furnaces.

New Cb-base alloy

Haynes Stellite Co., div. of Union Carbide Corp., recently announced the availability of a new, high-strength columbium-base alloy containing tungsten and zirconium. The alloy, designated *Cb-752*, is said to be one of the strongest materials of its type at temperatures above 2000°F. The alloy can be fabricated by conventional methods including welding, and has an ultimate tensile strength of over 25,000 psi at a temperature of 2400°F. The new alloy is expected to find use in aircraft and missile applications.

LD at Rotor plant

Ilseder Huette, Peine—one of the two rotor steelworks in Germany—is reported to be planning a new oxygen steel plant with 75-ton (LD?) converters and a total capacity of 1 million tons per year. The plant will replace the existing basic bessemer plant, but the rotor plant will be maintained in operation.

Thermoelectric research

Franklin Institute laboratories recently announced that a long-range basic program on thermoelectric phenomena and materials will be intensified during the coming year. Sponsored jointly by 13 industrial firms, the research will be concerned with bismuth, tellurium, silicon, and many intermetallics in the range of most heating and cooling applications. The goals of the program are to derive and analyze the basic theory of the behavior of themoelectric materials, to do research on the physics of semiconducting compounds (particularly their thermoelectric and energy transport properties), and to develop more efficient thermoelectric materials.

Sponsors of the study are: A. O. Smith, Borg-Warner, Carrier, Consolidated Mining & Smelting (Canada), Chrysler, E. L. Du Pont de Nemours & Co., Electrolux (Sweden), Fedders, Fuji Denki (Japan), General Motors, Linde (Germany), Robert Bosch (Germany), and Texas Instruments.

REPORTER

REPORTER

Canadian beryl discovery

Low-grade beryl ore, in substantial amounts, has recently been discovered in Manitoba, B. C., near Bernic Lake. The deposits, found on property owned by Chemalloy Minerals, Ltd., are estimated at approximately 600,000 tons of beryl ore which contain about 1.8-million lb of BeO. Chemalloy is reportedly planning to erect a concentrator to process the ores utilizing a Canadian engineered flotation process. The output is expected to be sold to Beryllium Corp., Brush Beryllium, and other US companies. For additional beryllium news, see p. 598.

Ugine-Sejournet extrusion

Battelle Memorial Institute has recently been granted a research license for the patented Ugine-Sejournet steel extrusion process, which uses molten glass as the lubricant. Under terms of the agreement, Battelle will use the process to conduct research for industry and government on various extrusion applications.

In the Ugine-Sejournet process, glass discs are placed in the extrusion container between the hot billet and the cold die. As the press ram moves the billet against the disc, a thin layer of glass melts and adheres to the billet; thus, the metal passing through the die is continuously coated with a thin film of lubricating glass. The glass lubricant makes it possible to extrude metals at temperatures far beyond the temperature limitations of conventional extrusion equipment. The method has met with success in the steel extrusion industry (see JOURNAL OF METALS, March 1961, p. 211), and it is hoped that it will be practical for the extrusion of high-temperature metals. Already, successful extrusions of molybdenum and tungsten have been made by the process at temperatures above 4000°F.

Al-Be rocket fuel

There is a good possibility that a mixture of granular aluminum and beryllium may soon be the principal ingredient for solid rocket fuels. It was pointed out recently that beryllium has a higher heat of combustion per unit weight than any other fuel element except hydrogen; and if compared on the amount of heat released per unit weight of fuel plus oxidizer, beryllium is superior to other elements including hydrogen. A disadvantage of the beryllium fuel is its toxicity, and this is expected to limit the use of the fuel to upper stage rockets.

BF coal injection

Injection of coal into blast furnaces is to be tried at Stanton Ironworks, Nottingham, England. A spokesman for the National Coal Board stated that coal injection could eventually replace at least 30 pct of the coke used in the furnaces; this would reduce the cost of producing iron and make possible increased production.

CPDA research contracts

The Copper Products Development Assn. (CPDA) recently awarded four contracts for the development of new and enlarged markets for copper. (See JOURNAL OF METALS, Feb. 1961, p. 115). Alloyd Corp., Cambridge, Mass. will work on the development of a low-bulk, high-temperature insulation for copper wire. The second study is for new high-strength copper alloys for devices requiring the continuous or intermittent support of mechanical loads. This work will be conducted by the Southern Research Institute, Birmingham, Ala. The third project, co-sponsored by the Cast Bronze Bearing Institute, will be directed toward the collection of engineering data for inclusion in the *Cast Bronze Bearing Design Manual*. Franklin Institute research laboratory will carry out this project. Basic exploration of the structural and atomic properties of copper will be investigated at Ghent University in collaboration with the Belgian government. These projects will bring the cost of the 1961 CPDA research program to about $400,000 for a total of 16 laboratory programs.

New tool steel

A new steel has been developed which can be heat treated to tensile strength in excess of 350,000 psi. Even at this high strength level, the material retains ductility and impact strength. The alloy was designed for tools and dies that require resistance to extreme shock. As an ultra-high strength steel, the new alloy is recommended for critically stressed components where maximum strength is required with the least sacrifice in toughness. Other advantages of the steel, made by The Carpenter Steel Co., Reading, Pa., include exceptional toughness, good machinability, low hardening temperature, and the subsequent benefits of simplified heat treatment. Called *Hi Shock 60*, the steel has unnotched Charpy impact strength of 110 ft-lb at 59 R_c hardness.

Type analysis of the patented *Hi Shock 60* is 0.68 pct C, 0.50 pct Mn, 0.30 pct Si, 1.00 pct Cr, 0.50 pct Ni, 1.00 pct Mo, 2.50 pct Cu, and 0.15 pct V. Specific gravity of the steel is 7.85. Tools and dies made from the new steel can be air hardened by heating uniformly to a temperature of 1600°F, depending on the size. For the best combination of hardness and toughness with minimum size change, tools can be tempered at 300° to 400°F.

The new steel also offers a good fatigue resistance. It has proved superior, in several applications, for punches used in cold extruding steel, and other punches used for some deep drawing or deep dimpling operations where flexing can occur. Other applications include: gun barrels, die and fixture used in hot tensile impact testing machine, die for forming reamer type blade for jack knives, and tool for making nail marks on jack knife blades. It has been used for applications such as hobs, heavy duty punches, mandrels and other tools and dies subject to high shock or fracture in service.

METALGRAMS

. . . news of "Electromet" ferroalloys and metals

UNION CARBIDE METALS

SEPTEMBER 1961

CHARACTERIZATION OF FERROALLOYS — To provide more complete information to steelmakers, UCM recently published data on the occurrence of hydrogen and non-metallic inclusions in alloy additives. Laboratory and field studies made over a period of many years demonstrate that these raw materials, which are used to control the composition of steel, exert a negligible influence, if any, on the gas content and inclusions present in steel. For details, write for the article, "Alloy Quality Versus Steel Quality," in the Summer 1961 issue of UNION CARBIDE METALS REVIEW.

* * *

TUNGSTEN -- HELPS ALLOYS RESIST HEAT -- Because of tungsten, high-speed steel and non-ferrous tools have cutting edges that stay hard under extreme friction and high temperatures. Hot-work tool steels punch, shear, or form metals at high temperatures without softening. Iron- and cobalt-base turbine blades, afterburners, and tailpipes resist erosion and heat from fast-moving hot gases. Union Carbide Metals supplies ferrotungsten, self-reducing tungsten, scheelite, and tungsten metal powder for such additions. Your UCM representative can give you further information. Also, ask for the article, "The Elements That Surround Us: Tungsten," in the Winter 1960 issue of UNION CARBIDE METALS REVIEW.

* * *

MANGANESE IN ALUMINUM -- In binary alloys, manganese improves the strength of aluminum by 20 per cent with no sacrifice in corrosion resistance. Thus, 3000 series alloys (containing 1.25 per cent manganese) are good general-purpose alloys for storage tanks, piping, furniture, TV antennas, awnings and siding, and chemical equipment. Manganese is also added to 2000, 5000, and 6000 series alloys to improve corrosion resistance. UCM offers low-iron ferromanganese and "Elmang" electrolytic manganese for such additions. More information can be obtained by writing for the article, "Manganese Puts Muscle in Aluminum," in the Winter 1961 issue of UNION CARBIDE METALS REVIEW.

* * *

SALUTE TO STEEL FOUNDRIES -- In 1861, the first steel castings made in the United States were poured in Buffalo, N. Y. They were railroad castings, still an important use of steel castings today. This year, steel foundries observe their first 100 years of serving the railroad, earth-moving and construction equipment, rolling mill, mining equipment, military, oil and gas, automotive, and other industries. Some 275 foundries in the U. S. and Canada can pour about 2 1/2 million tons per year for these uses. Union Carbide Metals wishes the steel foundry industry continued growth and success and looks forward to serving its ferroalloy needs in the future. Your UCM representative is always at your service.

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Topaz Mountain in Utah

WHAT'S HAPPENING TO BERYLLIUM?

Everybody is in the act developing new beryllium deposits and techniques for extracting the metal from non-beryl ores. This article attempts to unconfuse some of the confusion.

Amidst the mounting, and often conflicting, claims of new beryllium deposits, concentrating processes, and extraction techniques, two facts stand out: first, the United States is becoming self-sufficient in beryllium ores; and second, for the first time the industry is beginning to make use of ores other than beryl. The first fact is closely related to the second in that most of the beryllium deposits presently being developed in the United States are of non-beryl ores.

Substituting domestic for foreign ores could be of economic importance to the beryllium industry by permitting metal producers to have greater control over their sources of raw material. But the true significance of this development lies in the strategic importance of such a change, particularly in view of the extensive utilization of beryllium for military purposes.

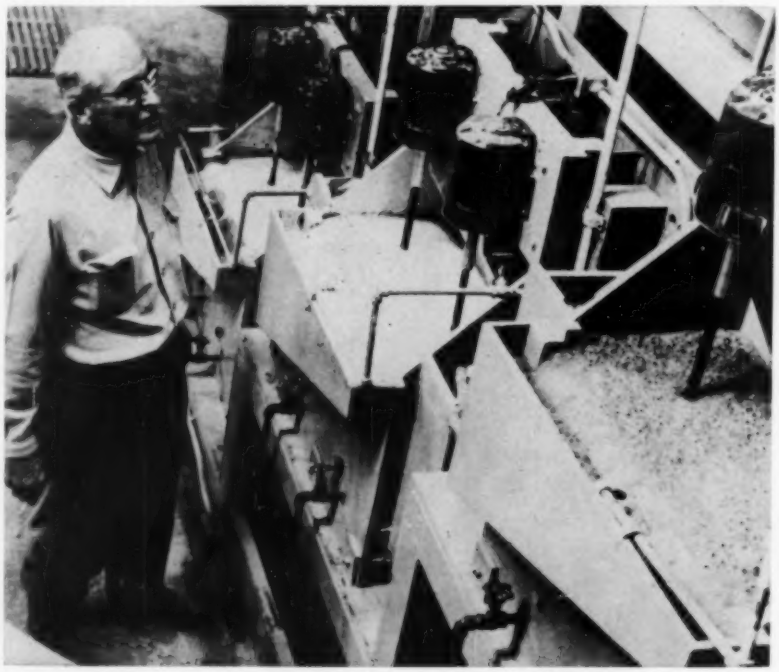
At present there are some 170 beryl mining operations (many of them small scale) in eight states with the major part of the output being from South Dakota, Colorado, and Utah. The largest producer is the U.S. Beryllium Corp. which operates the Boomer and Redskin mines at Badger Flats in Colorado. But, while originally beryl mines, they are also reported to be producing bertrandite ores. Engineers are presently studying the Boomer mine from the standpoint of open-pit operations.

The Carolinas have been in for their share of attention by beryllium enthusiasts, and Lithium Corp. of America, in conjunction with Alloyd Corp., expects to have a small beryllium pilot plant operating before long; it anticipates building a plant to treat purchased ores at Bessemer City, N.C. For several years the US Bureau of Mines, in cooperation with Foote Mineral Co., has been operating a flotation pilot plant using the Kings Mountain, N.C., spodumene-beryl ores; operations were described in MINING ENGINEERING, July, 1961, pp. 706-708.

Considerable attention is being given to development work being done by the Anaconda Co. on a phenakite deposit at Mount Washington in White Pine County, Nevada. But the area getting the most attention today is the Topaz Mountain deposit of bertrandite in west-central Utah. The deposit is reported to have some 3 million tons of 0.75 pct BeO ore. It is claimed that this deposit, being developed by several companies, will make the US self-sufficient in beryllium. But even if this claim fails to materialize, it is clear that the United States is making rapid strides toward a beryllium industry freed from its present dependence on foreign ore supplies.

The second factor—commercial utilization of non-beryl ores— can be of far-reaching economic importance by providing an extended raw-material basis for the industry. This should create greater consumer confidence in the metal which could provide for expansion of the industry and make for the development of lower-cost mass production techniques.

In addition, the extraction of beryllium from such non-beryl ores as bertrandite is likely to be a lower cost operation, although separation of impurities is more complex and will require new or modified techniques. Extraction from beryl (a beryllium aluminum silicate with 14



A. B. Campbell, metallurgical engineer in charge of Beryllium Resources' pilot plant, checks recleaner cells.

pct BeO) requires melting and quenching the ore or the utilization of a complex sintering operation in order to render beryllium amenable to acid or water leaching, respectively (JOURNAL OF METALS, Oct. 1960, pp. 793-797). But bertrandite, the hydrated silicate mineral containing 40 pct BeO, is, in some cases, amenable to sulfuric-acid leaching without prior treatment.

Another important economic factor derives from the fact that the non-beryl minerals, bertrandite and phenakite, contain 40 pct and more BeO as compared with 14 pct BeO for beryl. And finally, in many cases, the deposits of the non-beryl ores may permit lower-cost open-pit mining operations.

Possibilities of flotation

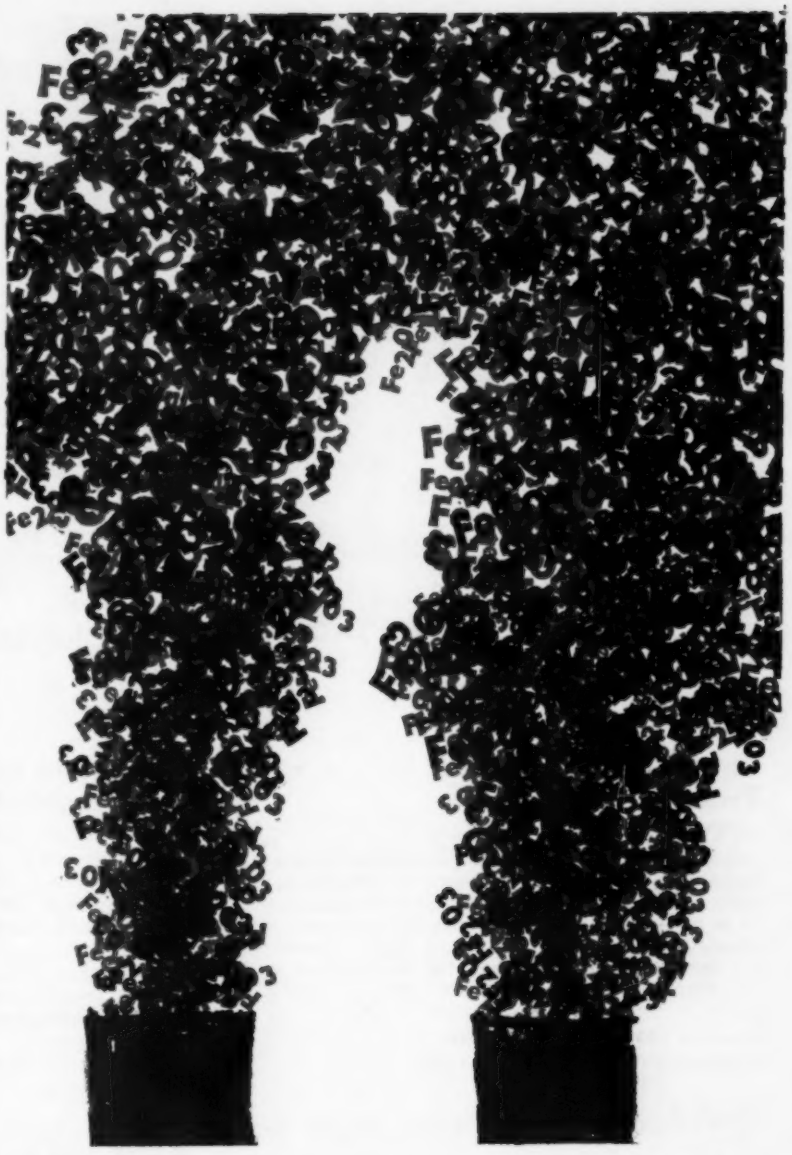
Another economic factor in the utilization of these non-beryl ores is the wide dissemination of beryllium values throughout known deposits. Beryl generally occurs in distinct crystals which can be hand cobbled to a 10 to 12 pct BeO mine product, but this is not possible with the non-beryl ores. Even with beryl mining the need was felt to develop an ore concentrating technique which could by-pass the labor-expensive hand cobbling, and for non-beryl ores such a process was an essential step in their utilization. It appears that at least three beryl flotation processes have been developed: one by the USBM for the Kings Mountain beryl-spodumene ores of North Carolina, a second by the Mineral Concentrates & Chemical Co. (Mincon) which is in commercial operation at Loveland, Colo., for treating ores from Badger Flats mines; and the third a process successfully developed by E. Van Dornick.

The adaptation of flotation to the concentration of the non-beryl ores has been successfully pursued, in at least two instances. Mincon claims to be treating bertrandite and beryl ores by a "new inexpensive water flotation process pioneered by Mincon". And the firm is now constructing a concentration plant in Colorado near the Boomer mine of U.S. Beryllium. Scheduled for completion in September, the mill is designed to treat 100-tpd of 3 to 4 pct BeO ores. Mincon claims that it can take a 1-pct BeO ore and economically produce concentrates containing from 15 to 18 pct BeO. It has supplied Beryllium Corp. with batches of 28-pct concentrates.

Van Dornick and Topaz

The most publicized flotation process for non-beryl ores has been that developed by E. Van Dornick at the Beryllium Resources pilot plant in Los Angeles. After having successfully developed reagents for floating beryl, the pilot plant moved on to

(Continued on page 601)



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edited by **GEORGE R. ST. PIERRE**

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Extractive Metallurgy of Copper, Nickel, and Cobalt

Based on an International Symposium sponsored by the Extractive Metallurgy Division of The Metallurgy Society of AIME; New York, February 15-18, 1960

edited by **PAUL QUENEAU** including an Annotated Bibliography by **Ken G. Robb**

CONTENTS ***(Showing only Session titles: A total of 24 papers were given, each followed by discussion.)***

General — Fuel-Fired Smelting and Converting — Electric Furnace Smelting — Atmospheric and Elevated Pressure Leaching — Refining — Bibliography

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Response of Metals to High Velocity Deformation

Volume 9 of Metallurgical Society Conferences

Sponsored by the Physical Metallurgy Committee, Institute of Metals Division, The Metallurgical Society of AIME, Estes Park, Colorado, July 11-12, 1960

edited by P. G. SHEWMON and V. F. ZACKAY

CONTENTS

High Velocity Deformation (6 papers)—Shock Phenomena in Metals (10 papers)

May, 492 pages **Members: $14.40, Nonmembers: $18.00**

A complete listing of forthcoming and previously published volumes is available from The Metallurgical Society or Interscience Publishers upon request.

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published for The Metallurgical Society of AIME by INTERSCIENCE PUBLISHERS, 250 Fifth Ave., N.Y. 1

Beryllium (Con't)

the phenakite ores of Mt. Wheeler in Nevada and likewise achieved success. But the biggest problem was yet to come—floating the complex ores from the Topaz Mountain region of Utah.

In this ore, the beryllium compound appears to be hydrated and amorphous. It is quite definitely associated with three other minerals, likewise amorphous in this occurence: pyrolusite, fluorspar, and chalcedony or opal. The degree of solid solution of the beryllium oxide in the three varies from 0 to 15 pct and averages about 3.5 pct. Since the gangue is an altered rhyolitic tuff containing considerable amounts of bentonitic-type clays, the most serious problem in the flotation of these ores is the *sequestering* of those clays that do not contain appreciable amounts of BeO. This is not easy, since the flotation of one group of amorphous minerals from another is one of the most difficult separations known.

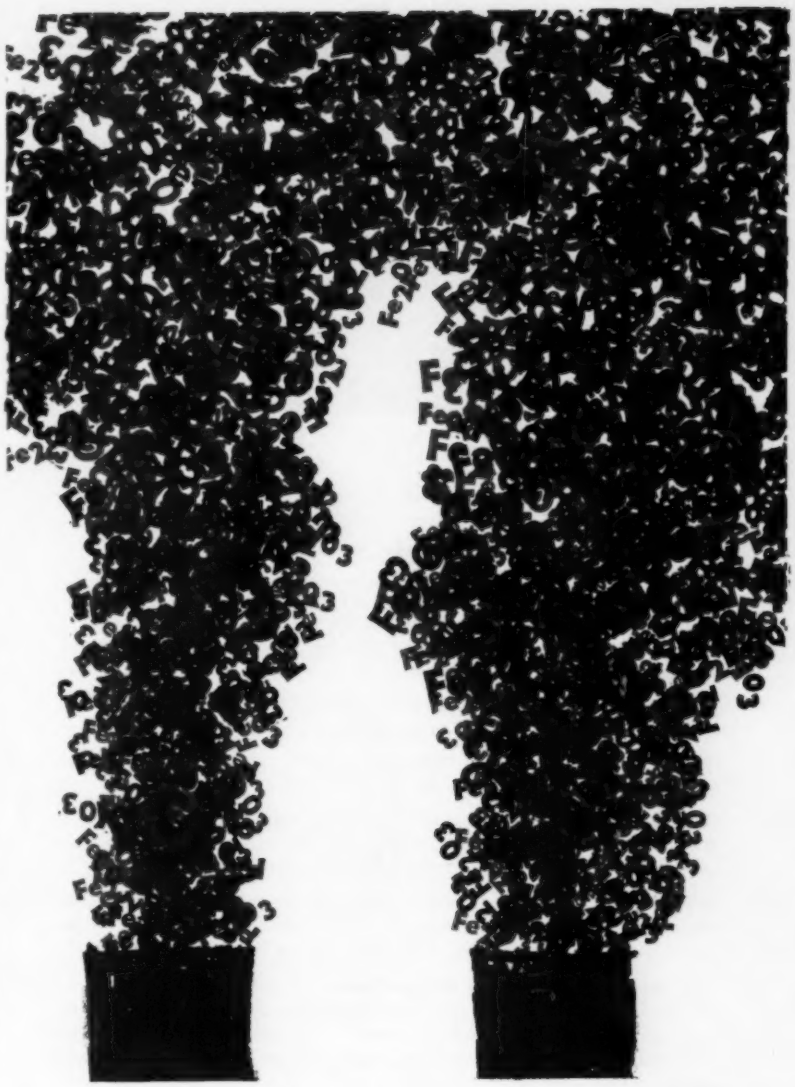
Dr. Van Dornick describes the problem himself: "In the case of Topaz Mountain ore the problem of flotation is greatly complicated both on a theoretical and actual basis. This is due to the fact that the beryllium mineral is not present in an apparent identifiable crystalline form but instead is in a heterogeneous occurrence of gradations from definite chemical compositions through pseudo crystalline forms down to solid solutions in the host gangue.

"Flotation concentration is basically dependent upon a concentration of the desired mineral in a discrete particle, and the lack of such a particle theoretically make concentration impossible. Fortunately, in the case of Topaz ore, there is enough segregation of the beryllium mineral to permit appreciable concentration by flotation, but the problem was extremely difficult. Not only was it difficult due to lack of particle existence, but it was greatly complicated because of the clay character of the ore body. Adequate grinding to liberate the beryllium values resulted in excessive slimes production and chemical leaching.

"Inasmuch as such slimes also contained beryllium values, they could not be discarded but instead had to be processed to permit recovery of their value. A substantial chemical technique of slimes handlings by dispersants, sequestrants, pH control plus the usual mechanical procedures was necessary in dealing with the slimes problems.

"Even after isolation of the slimes problems, the Topaz ore was still difficult due to its lack of segregated beryllium values and required the most carefully discerning chemical and pilot-plant analysis of the conditions necessary for a satisfactory concentration of the desired minerals."

(Continued on page 603)



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SUBJECT:

silicon

Its behavior in quality steels

Silicon is one of the most common alloying and process elements used in steel. There are three properties of Si utilized in steel production.

(1) Silicon is a moderately powerful deoxidizer.

(2) In quenched and tempered, or pearlitic steels, it increases strength.

(3) In low carbon steels, it increases electrical resistivity and is an aid to formation of large, well-crystallized ferrite grains.

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For more details on Foote Electromanganese®, write for Bulletin 201, Foote Mineral Company, 424 Eighteen West Chelten Ave., Phila. 44, Pa.

FOOTE
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personals

William M. Baldwin, Jr., research professor of metallurgy at Case Institute of Technology, has received an $18,000 research contract from the American Iron and Steel Institute for a study of brittleness of non-sensitized stainless steels at high deformation speeds.

William H. Bickley, technical information specialist, has joined the staff of Clyde Williams & Co., Columbus, Ohio. He was formerly with Battelle Memorial Institute.

George A. Brunner has been made assistant district sales manager of the Pittsburgh district of E. J. Lavino & Co. He has been with the company since 1924. **Harry J. Hausner** is now assistant district sales manager in the Chicago district. He has been a salesman since 1951.

Harry Czyzewski, manager of Metallurgical Engineers, Inc., Portland, Ore., has been awarded a certificate of appreciation by the Consulting Engineers Council. He is a member of the group's board of directors.

Falih H. Darmara is president of Special Metals, Inc., a newly formed company which recently purchased the Kelsey Hayes Metals div. of which he was president and general manager. **Harry J. Hamjian** and **Italo S. Servi** are vice presidents of Special Metals; they were formerly manager of plant operations and technical director, respectively, of the recently purchased metal div.

John V. N. Dorr has resigned from the board of directors of Dorr-Oliver, Inc. He had also been honorary chairman of the board.

George R. Eusner has been elected vice president of the Davis Fire Brick Co., at Oak Hill, Ohio. He was formerly chief of the refractory and mineral technology div. of U.S. Steel Corp., at Monroeville, Pa.

Christopher J. B. Fincham has been appointed technical director of the Metals div. of National Research Corp. He joined the company in 1956.

Ewan W. Fletcher of Massachusetts Institute of Technology has been named director of a basic research program with primary emphasis on solid state physics of metals which will be launched by Kennecott Copper Corp.

Antoine M. Gaudin of Massachusetts Institute of Technology has been re-elected chairman of the Engineering

(Continued on page 609)

Beryllium (Con't)

But in spite of the difficulties, the results of the work at the Beryllium Resources' pilot plant have been reported to be successful. Dr. Van Dornick has stated, "We succeeded in making a good ratio of concentration on the Topaz clay with reasonably high recovery by judicious control of the grinding, pulp density, and pH. . . . The quantity and point of application of the reagents is critical. Last, but not least, the beryllium coatings formed are sensitive to mechanical impregnation under violent agitation. This is overcome by the use of special porous-diaphragm air diffusers and very light agitation in the flotation cells."

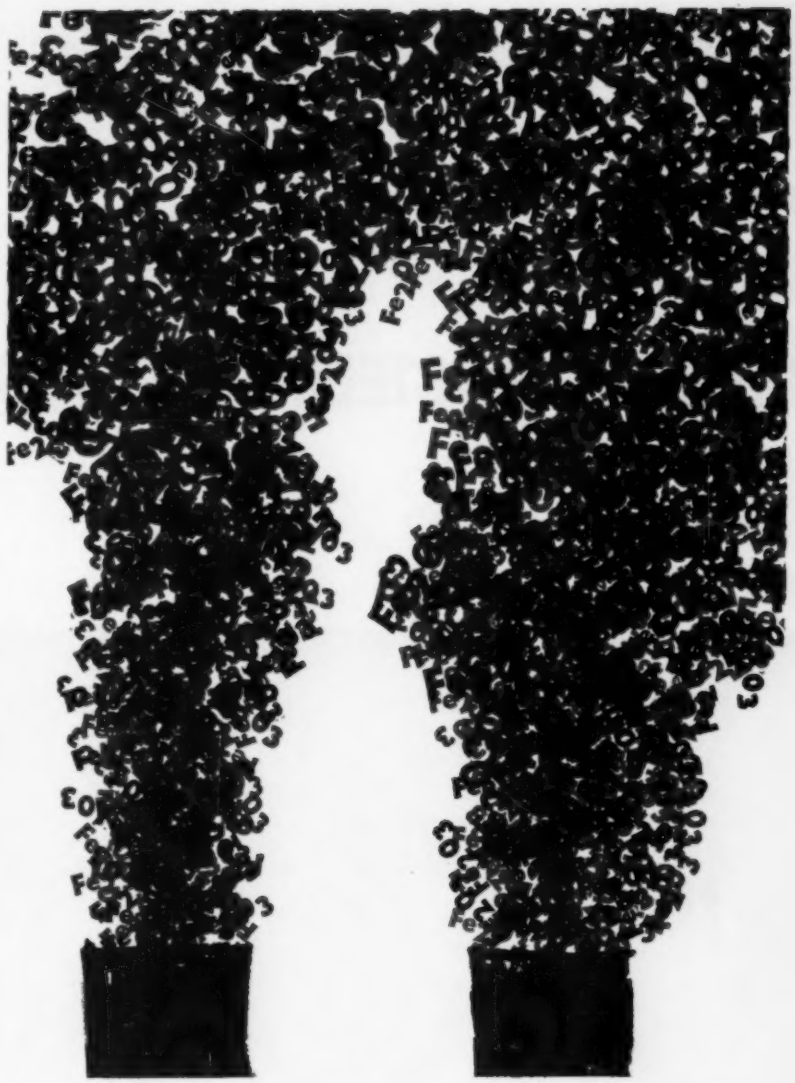
A flow sheet of the process accompanies this article; in its general features it follows conventional flotation practices. Reagents take advantage of the amphoteric nature of beryllium and its ability to form organic compounds with chelating-type groups to give a start on which to build stable water-repellent and air-avid flotation coatings specific to beryllium atoms. But the exact nature of the reagents is a closely-guarded secret, and Van Dornick has set up a separate firm to handle them. It is not intended to apply for a patent on the process, and for this reason, the secret must be maintained. The grade of the concentrate produced from the 0.75-pct BeO Topaz ores has not been disclosed, but it can be said that greater emphasis is being put on the efficiency of beryllium recovery, and this is reported to be about 85 pct.

Based on the success of the pilot-plant work Beryllium Resources has decided to construct a 250-tpd flotation plant in the vicinity of Delta, Utah, but construction work has not yet begun.

In the meantime, the Beryllium Resources' pilot plant in Los Angeles is doing work on the flotation of Brazilian beryl prior to starting on a completely different phase of development—carrying through on a pilot-plant scale, the chemical techniques for extracting beryllium hydroxide from the concentrate. These techniques are said to have been successful on a laboratory scale. The eventual goal would be to produce a 95-pct beryllium-hydroxide product at Delta, Utah.

Beryllium Resources' associate, Brush Beryllium, has been working with Topaz ores and concentrates supplied by the Los Angeles pilot plant. And the Brush pilot plant at Elmore, Ohio, has reported to have developed its own techniques for extracting beryllium hydroxide. How close the collaboration is between Brush and Beryllium Resources on this point is difficult to say. Perhaps the Beryllium Resources' pilot plant will carry forward Brush's work. It would certainly appear more eco-

(Continued on page 606)



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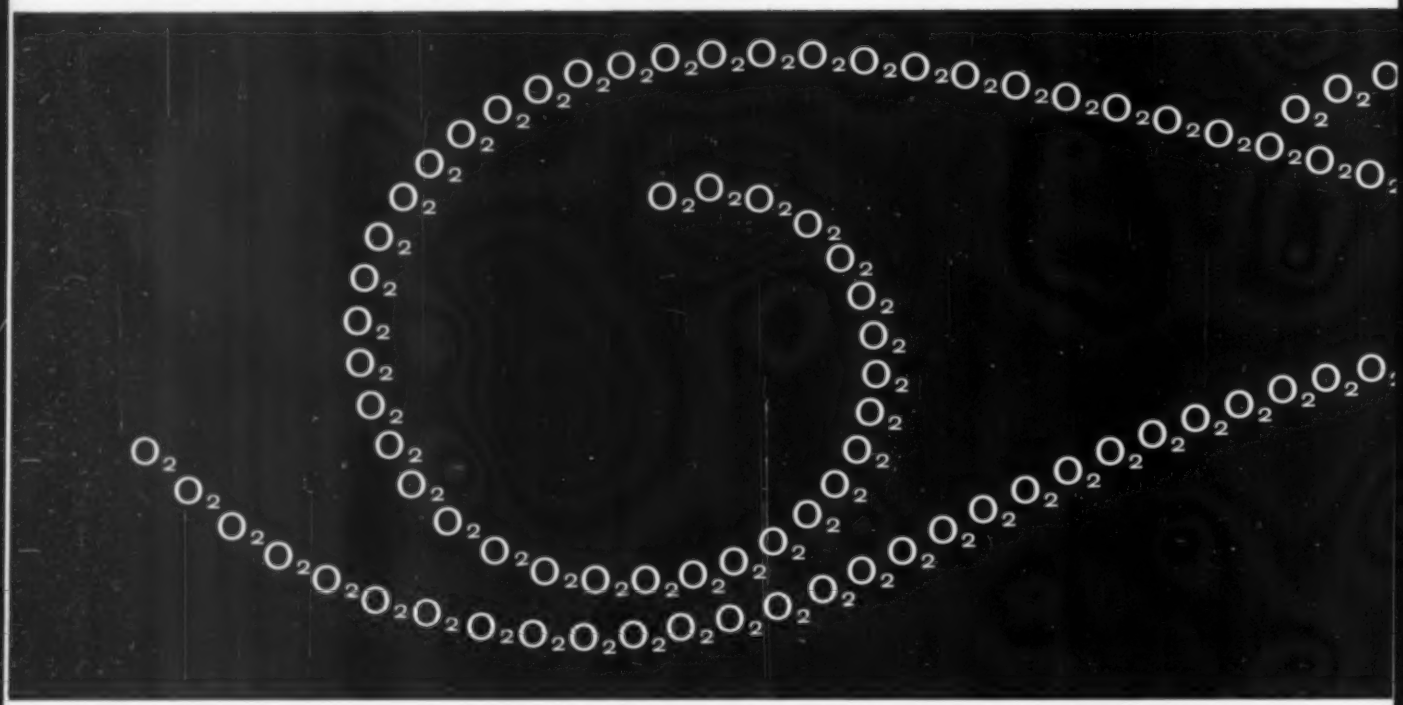

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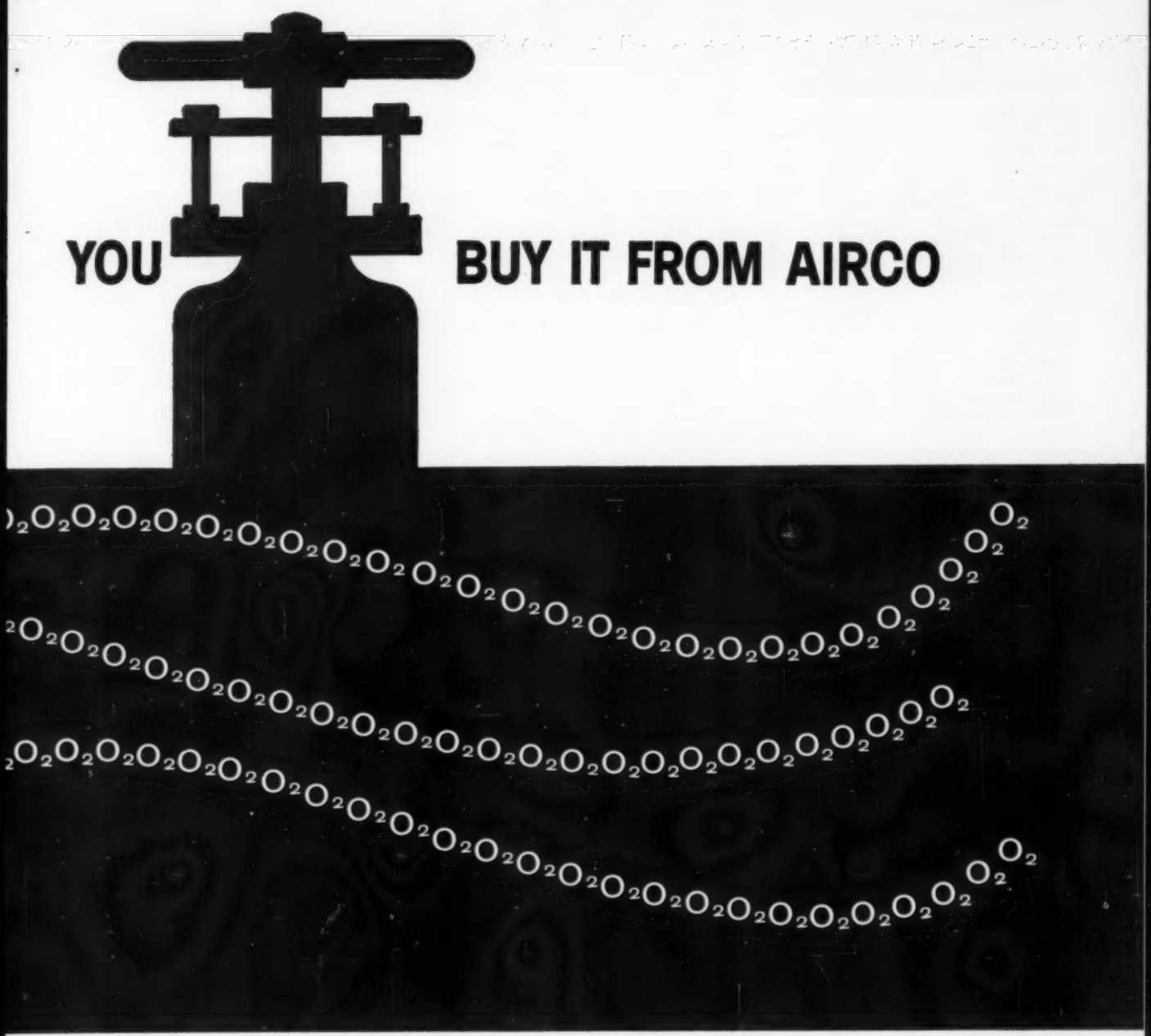

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nomical to produce the hydroxide product close to the deposit in order to save on transport costs.

UTI and direct extraction

Although Beryllium Resources is not the only firm which has been pursuing the problem of chemically extracting beryllium values from Topaz Mountain ores, it is the only film which has been attempting to use a combination of flotation and chemical extraction. The other firms have apparently been pursuing the course of direct extraction. Capital costs for such a course might be lower, it is true, but another factor which must be taken into consideration is the absorption of leaching agents by the clay-like ores.

The company which is furthest advanced in direct extraction is United Technical Industries (UTI). This firm has claims in the Topaz Mountain area from which they have been mining and processing fluorspar on a small scale for some time. With the discovery of beryllium values on the property, ". . . UTI undertook a crash program of research in June of 1960 to develop processes for the utilization of the new ores." The firm has been operating a pilot plant at Murray, Utah, and there is reason to believe that they are now in the process of reconverting their fluorspar mill to beryllium operations. The firm expects to begin the shipment of beryllium oxide before the end of the year. Capacity of this acid-extraction plant has been stated to be 25,000 lb of 97-pct BeO per month. The economics of the operation are closely tied to acid consumption, which is reported to be ½ ton of acid for 1 ton of ore.

Other firms active in the Topaz Mountain area are Vitro, Combined Metals, and Food Machinery. The first of these, Vitro has carried out experimental work and developed a leaching technique for the ores. Food Machinery is also believed to be actively engaged in this same pursuit.

On the metal side

Expanded facilities are not limited to a wider source of raw materials, for both of the US beryllium metal producers are engaged in expansion programs.

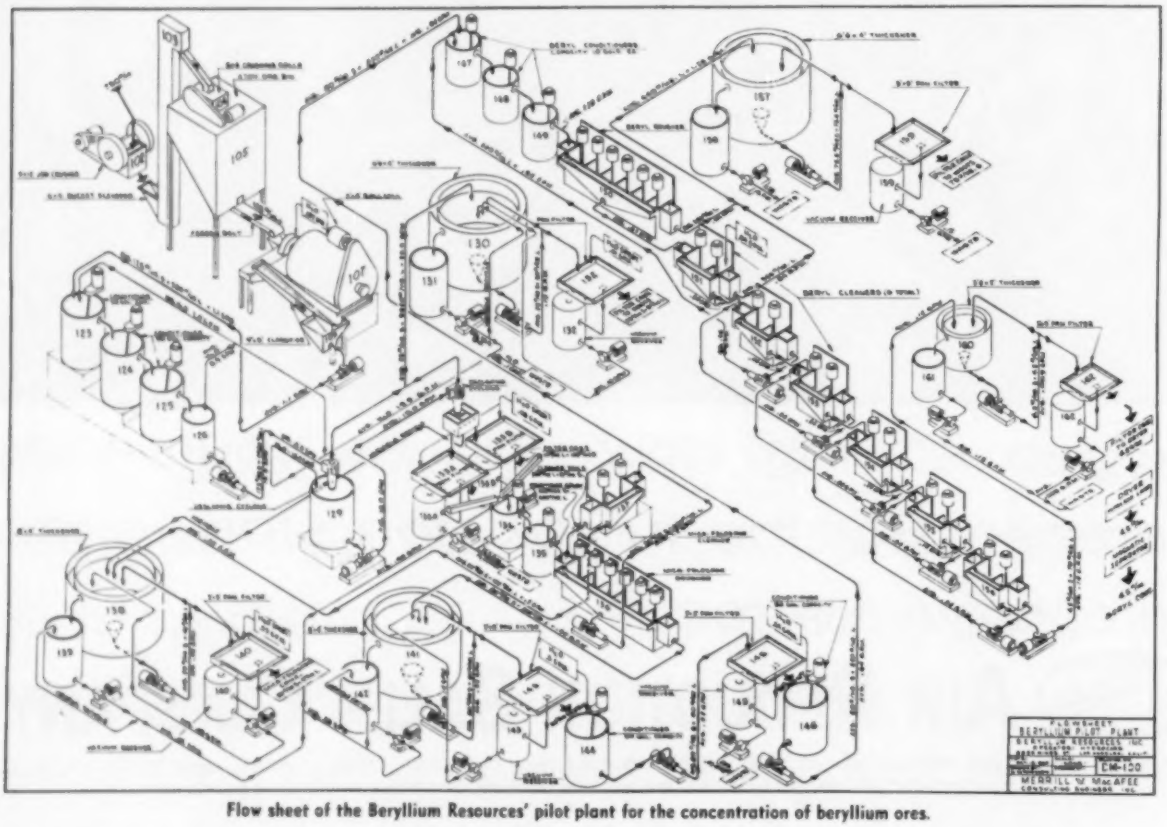
The Beryllium Corp. (Berylco) has completed a $3.5-million expansion of its Hazleton, Pa., plant, boosting its metal capacity by 150 pct to 25,000 lb per month. Expansion included both billet plant and fabricating facilities. And as a part of an overall expansion at Brush Beryllium Co., a $3-million plant designed to increase the firm's fabricating capacity by 50 pct has started production in Cleveland. When completed later this year, the facility will replace three small fabricating units in the same city. At Elmore, Ohio, a $6-million expansion program will double capacity to produce the metal required for Brush's machining operations in Cleveland. The result of these expansion programs is that capacity to produce beryllium metal in the US is reportedly 55,000 lb per month.

Conclusions

Apart from the two conclusions drawn at the outset of this article—namely that the US is to become self-sufficient in beryllium ores and that the industry will start using ores other than beryl—the only other conclusion that might be drawn is that the net result of the many developments taking place in the industry will tend to make for lower cost beryllium, hence a larger market for the metal. Both of the major metal producers—Brush and Beryllium Corp.—have secured for themselves a position in the Topaz Mountain development. But mining and beneficiation activities are being pursued elsewhere, particularly in Colorado and in North Carolina. Under these conditions it is inconceivable that the present metal producers can remain the only ones in the field.

To the specific question of whether the Van Dornick flotation process (followed by acid extraction) or the UTI direct acid extraction process will prove to be the most economic, we shall let time provide the answer.

F. Weston Starratt



Flow sheet of the Beryllium Resources' pilot plant for the concentration of beryllium ores.

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editorial

Basic Oxygen Steelmaking —A Landslide

Since 1952, talk among steelmakers has increased from a whisper to a thunderous roar about the new basic oxygen steelmaking processes. The LD process was generally greeted with skepticism on all sides, for after all, it was developed as an answer to the specific technical and economic problems facing the Austrians in their efforts to rebuild and expand their steel industry during the desperate post-war years. Nevertheless, many engineers went to Linz and Donawitz to look and ask questions. Dominion Foundries & Steel Ltd. at Hamilton, Ontario, Canada, was the first firm to take the Austrians up on the new steelmaking process by putting into operation their LD basic oxygen plant in August 1954. Other firms throughout the world began to follow suit. But still others were at work in their laboratories and pilot plants on different approaches to basic oxygen steelmaking which eventually turned into such processes as the Rotor, Kaldo, LD-AC, and the OLP.

The result has been a landslide toward basic oxygen steelmaking. A recent international study concluded that no new acid bessemer shops will be built, that there is little probability that new bottom-blown bessemer shops will be built even for blowing non-nitrogen-containing mixtures, and that hardly any new open-hearth shops will be built, but that basic oxygen steelmaking installations and electric-arc furnaces will be built exclusively in most new steel shops. In major steel producing countries, the main emphasis will be on the installation of oxygen furnaces of the LD and Kaldo types. For example, in France between 1960 and 1965, the capacity of oxygen vessels of the LD and Kaldo types will be increased by some 6 million tons and that of electric furnaces by 1 million tons. During this same period there will be no increase in open-hearth capacity and even a slight decrease in the capacity of basic bessemer converters. This same trend is true for the other countries of the European Coal and Steel Community and Japan, although the United Kingdom anticipates some increase in open-hearth capacity along with the basic oxygen installations.

In the United States the trend toward the basic oxygen steelmaking process is fast becoming a landslide. At the beginning of this year, there was some 4¼ million tons of capacity, and this figure will be doubled by four new basic oxygen steel plants: three of the LD-type—one brought into operation in July, one scheduled to go on stream at the time of this issue, and the third in the advanced stages of construction—and the fourth plant to be this Country's first Kaldo installation which is now under construction.

We dedicate this issue to the foresighted engineers responsible for these four modern plants which incorporate many new design features. Congratulations to all concerned! FWS

Personnel

(Continued from page 584)

Engineers. (b) Metallurgical Engineer, graduate, experienced in heat treating, surface treatment, i.e., plating and electroplating of special steels. $9000-$13,000. (c) Chemical-Metallurgical Analyst experienced in the use of infra-red spectrophotometry and chromatography. Spectrographic experience helpful. $8000-$13,000. New York State. W518.

Metallurgist-Steel: $12,000-$15,000, in Illinois, Met.E., Chem.E., M.E. Background in metallurgy, chemistry, electricity. Develop new steels for laminations in conjunction with steel mills, control application of silicon steel to laminations, improve stamping quality, review and set annealing procedures. Become familiar with electrical and magnetic requirements of product in order to develop characteristics of steel tailored to product. If possible with chemical background, assist in plastics and paint problems. Sj-6260.

Junior Mining, Metallurgical-Mill, Mine: $375 plus room and board and traveling, liberal vacation, retirement plan, 10 pct bonus, two weeks vacation after one year, six weeks after second year, company will pay foreign income tax, in Central America, recent graduate, single status, some knowledge of Spanish. For expansion of existing mill and mine facilities, possibly later into production. Sj-6275.

Quality Control Engineer-Food Containers: about $700, in Stockton, graduate Chem.E., Met.E., age to 35. Preferably experienced in quality control (laboratory, plant, process) in high speed, mechanized can making for a foods plant. Should be knowledgeable about plating, coating, sealing. Able to carry out laboratory work, set up standards, install quality control procedures, provide continuing assurance of controlled processing and end product. For a manufacturer of metal cans for food processing. Sj-6159.

Books

(Continued from page 589)

the specific problems for which they offer solutions.

The second part of the book offers a detailed treatment of specific problems of temperature measurement (organized by situations), and the techniques most suitable for solving them.

Metal Statistics, 1961, edited by Norbert J. Langer, published by American Metal Market, 829 pp., $3.50 ($3.75 if billed), 1961. •

This is the 54th edition of this yearbook which contains more than 400 pages of statistics covering metals from aluminum and beryllium to vanadium and zinc. The volume also contains an index, a 130 page Buyers' and Sellers' Guide, and numerous advertisements. A number of new tables have been added to the book.

Copies of the volume may be obtained from American Metal Market, 18 Cliff Street, New York 38, N. Y.

ASM Review of Metal Literature, 1960, published by the American Society for Metals, vol. 17, edited by Marjorie R. Hyslop, 1342 pp., $25.00, 1961. •

This volume contains 11,903 annotations of articles, technical papers, reports, and documents appearing in engineering, scientific, and industrial journals and books throughout the world during 1960. The book is divided into 20 sections covering different areas of materials and process engineering and technology, and includes addresses of publications, as well as comprehensive subject and author indexes.

Back issues of volumes 1 through 16, 1944-1959, also are available at $20.00 per copy.

Handbook of Thermophysical Properties of Solid Materials, prepared under the direction of Alexander Goldsmith, Thomas E. Waterman, and Harry J. Hirschhorn, Macmillan Co., five volumes, 4300 pp., $90.00, 1961. •

This handbook was prepared originally by the staff of Armour Research Foundation for the exclusive use of Air Force contractors in aircraft, missile, space, and nuclear science fields. It contains all data published over a 17-year period on 12 vital thermophysical properties of solid materials that melt above 1000°F, including elements, alloys, ceramics, cermets, polymerics, and composite materials.

Five properties of the 12 covered —density, melting point, latent-heat of fusion, latent-heat of vaporization, and latent-heat of sublimation—are grouped together for convenience on a single sheet. The seven other properties—specific heat, thermal conductivity, thermal diffusivity, emissivity-reflectivity, linear thermal expansion, vapor pressure, and electric resistivity—are evaluated on separate data sheets and plotted on separate graphs as functions of temperature.

The investigator's method of testing is given for each entry. Special additional information includes the experimentor's own estimate of the accuracy of a test, and information on prior treatment of the material, or special environment of the test.

Powder Metallurgy in Nuclear Reactor Construction, by H. H. Hausner, International Atomic Energy Agency-Vienna, 72 pp., $1.00, 1961.

This review covers the general principles of powder metallurgy, the basic variables in its technique, and the characteristics and methods for fabrication of the powder particles, together with methods for fabrication of powders for nuclear engineering processes and principles for the safe handling of these materials. Special emphasis is given to the powder metallurgy fabrication of nuclear fuel materials; also information on powder metallurgy preparation of control materials, moderators, and construction materials. The latest data is given on fiber powder combinations, sinter welding, and sintering under irradiation.

Copies of the booklet may be ordered in the US from the National Agency for International Publications, Inc., 801 Third Ave., New York 22, N. Y., or from the United Nations Bookshop, U.N. Headquarters, New York, N. Y.

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Personals

(Continued from page 602)

Foundation, research department of United Engineering Trustees, Inc.

Russell B. Gunia, manager, stainless steel metallurgy, U.S. Steel Corp., has received the American Society for Testing Material's Award of Merit for outstanding service to the organization. **James H. Lansing,** executive secretary of the Ductile Iron Society of Cleveland, Ohio, also received an award.

Russell P. Heuer, vice president of General Refractories Co., will receive the Francis J. Clamer Medal of The Franklin Institute at the Institute's Medal Day ceremonies on October 18. He is receiving the award for his pioneer work in the composition and manufacture of basic brick and in the design of openhearth roof structures.

J. P. Hirth is now Mershon associate professor of materials science at Ohio State University. He was formerly assistant professor at Carnegie Institute of Technology.

A. L. Hodge of the Linde Co. has received the American Iron and Steel Institute Medal for his paper, which was presented at the Institute's 1960 General Meeting. The title of his paper was *Predicting Effects of Oxygen, Moisture and Fuel Additions on Blast Furnace Operation with Electric Computers.*

Walter P. Huhn, Jr., has been appointed manager of metallurgical services for American Potash & Chemical Corp., and will make his headquarters in Industry, Pa. He was formerly a senior melting metallurgist with Crucible Steel Co.

Alexander B. Kane has been appointed superintendent, and **Howard J. Bardwell** assistant superintendent of The Anaconda Co.'s slag treating plant at East Helena, Mont. Mr. Kane replaces **Robert L. Thompson,** who retired in May.

A. A. Kappenhagen has been named assistant district manager for Republic Steel Corp. at Warren, Ohio. He was formerly general superintendent of the company's Chicago district.

Thomas B. King has been appointed a full professor of metallurgy at Massachusetts Institute of Technology. He was formerly associate professor.

Joseph B. Kushner, professor of engineering at Evansville (Indiana) College, has been awarded a $8300 grant from the National Science Foundation to conduct research on electrodeposition.

R. A. Lewis has been named head of the reduction research section of Kaiser Aluminum and Chemical Corp., at Permanente, Calif. He was formerly research supervisor.

Gunvant N. Maniar is now a staff metallurgist with Crucible Steel Co. of America in Pittsburgh. He is with the applied physics group of the Central Research Laboratory, where he is working with the electron probe microanalyzer.

Norman A. Matthews has been appointed group leader, ferrous metallurgy, of the research laboratory of International Nickel Co., Inc., at Bayonne, N. J.

Bruce Orr is now manager of the newly formed tungsten sales div. of Climax Molybdenum Co. He was previously assistant sales manager of the company's steel div.

F. D. Rosi has been appointed assistant laboratory director of materials research at the RCA Laboratories in Princeton, N. J.

E. Scala, manager of the materials department at the Avco Corp.'s research and advanced development div., will be professor of metallurgy and materials sciences at Cornell University, effective September 1.

John Leo Scarry has been named assistant general superintendent, primary production, at the Riverdale plant of Acme Steel Co. He has been general superintendent of A. M. Buyers Co. since 1959.

Milton Stern has been appointed manager of research of Linde Co.'s Speedway, Ind. research laboratories. He has been assistant manager of research since 1959.

(Continued on page 651)

METALLURGIST

Ethyl Corporation is expanding its activities in metals and metal coatings via organometallic chemicals. To support its chemists in this expanded effort Ethyl seeks a metallurgist with three to ten years experience and with at least a B.S. degree in physical sciences or engineering. This responsible position, located at Baton Rouge, will involve evaluating metallurgical properties of and defining applications for metals and metal coatings obtained from organometallic compounds.

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Effect of Strain on Diffusion in Metals, by A. G. Guy and J. Philibert (IMD)

The Titanium Rich Region of the Titanium-Aluminum-Vanadium System, by Paul A. Farrar and Harold Margolin (IMD)

The Titanium-Zirconium-Oxygen System, by Michael Hoch, Robert L. Dean, Chung K. Hwu, and Samuel M. Wolosin (IMD)

Aging of Supersaturated Alpha Phase in a Copper-Silicon Alloy, by Gary A. Dreyer and D. H. Polonis (IMD)

The Creep Behavior of Heat Treatable Magnesium-Base Alloys for Fuel Element Compounds, by P. Greenfield, C. C. Smith, and A. M. Taylor (IMD)

Yield Points in αCu-Al Single Crystals, by T. J. Koppenaal and M. E. Fine (IMD)

Effects of Alpha-Soluble Additions (Aluminum, Carbon, Oxygen) on the Structure and Properties of Titanium-Molybdenum, by F. C. Holden, H. R. Ogden, and R. I. Jaffee (IMD)

Grain Refinement of Steel Ingots by Solidification in a Moving Electromagnetic Field, by Frederick C. Langenberg, Guenter Pestel, and C. Richard Honeycutt (ISD)

The Tantalum-Tungsten-Rhenium System, by J. H. Brophy, M. H. Kamdar, and J. Wulff (IMD)

Solid Solution Strengthening of Ag by Al, by A. A. Hendrickson and M. E. Fine (IMD)

Observations Concerning Zone Refining and Thermal Treatment of Molybdenum from Low Temperature Resistance Measurements, by E. Buehler and J. E. Kunzler (IMD)

On the Thermally Activated Mechanism of Prismatic Slip in Magnesium Single Crystals, by P. Ward Flynn, J. Mote, and J. E. Dorn (IMD)

The Effects of Deformation at 78°K on the Alloy Cu_3Au, by Barton Roessler and M. B. Bever (IMD)

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Thermodynamics of Silicon Monoxide (with an Appendix by P. J. Bowles,) by H. F. Ramstad and F. D. Richardson (ISD)

Structural Transformations in a Ag-50 at. pct Zn Alloy, by H. W. King and T. B. Massalski (IMD)

The Constitution Diagram Niobium (Columbium)—Rhenium, by Bill C. Giessen, Rolf Nordheim, and Nicholas J. Grant (IMD)

An Analysis of Powder Compaction Phenomena, by R. W. Heckel (IMD)

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The Tungsten-Oxygen System, by George R. St. Pierre, William T. Ebihara, Monte J. Pool, and Rudolph Speiser (EMD)

An Experiment on the Influence of Alloying Elements on the Internal Friction of Cold Worked and Quenched Martensitic Iron and Steel, by I. Tamura, T. Mura, and J. O. Brittain (IMD)

education

EJC Report

Recent achievements and future goals of the Soviet Union in the field of engineering, engineering education, and manpower utilization are presented in a report published by Engineers Joint Council, Inc.

The USSR annually graduates approximately 117,000 engineers compared to 38,000 for the US. Soviet plans call for substantial increases in that number.

The report, *The Training, Placement, and Utilization of Engineers and Technicians in the Soviet Union*, is based on a tour of the Soviet Union made last year by six representatives of US engineers. The tour was sponsored by Engineers Joint Council with financial support of the National Science Foundation, and took place under an exchange agreement between the US and the USSR. The 112-page document, including footnotes, statistical data, and photographs, is available in limited numbers for $1.00 each from Engineers Joint Council, 345 East 47th Street, New York 17, N. Y.

Illinois Institute of Technology

Construction has begun at Illinois Institute of Technology of a new John Crerar scientific library. The building will be of modern design consistent with neighboring Institute buildings. It is scheduled for completion in the late summer of 1962. The library is devoted to the sciences, engineering, and medicine.

The new building will be two stories in height with a total area of 92,160 sq ft.

M.I.T.

Massachusetts Institute of Technology has received a $3,275,000 contract from the Defense Dept.'s Advanced Research Projects Agency for materials research. The contract is one of five for a total of $13,375,000 made with five universities. The others are Stanford University, Harvard, Brown, and the University of Chicago.

Scientific objectives of the new program at M.I.T. will lie in four active fields considered likely to lead to important future results and likely to attract an increasing number of students. The four fields are: Magnetic and Low Temperature Research; Semiconductors and Their Applications to Devices; Electronic Materials Development and Preparation; and Solid State Structure Studies by Advanced Techniques.

The expanded materials research program will require additions to the M.I.T. staff, including faculty, post-doctoral workers, and graduates. More than 100 members of the M.I.T. faculty are now engaged in materials research and education.

University of Arizona

The nation's first formal educational program in scientific hydrology—the science of water—will be offered this fall at the University of Arizona. The new program will lead to the B.S., M.S., and Ph.D. degrees in scientific hydrology.

The University of Arizona program will offer six new scientific hydrology courses which will serve as the core of the graduate study program. The new courses are field hydrology, hydrologic systems, dynamics of flow systems of the earth, continental hydrology, analog model analysis of hydrologic systems, and mathematical statistics in hydrology.

Elementary courses in hydrology

(Continued on page 650)

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The International Nickel Co., Inc., Development & Research Division, seeks a graduate Metallurgist or Metallurgical Engineer with 5 to 10 years experience in ferrous metallurgy, particularly steel strip processing. Primary responsibility will be to initiate and carry out product development in metallic coatings on steel. Prior coating experience not essential.

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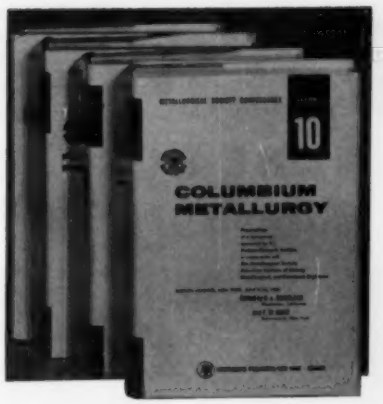

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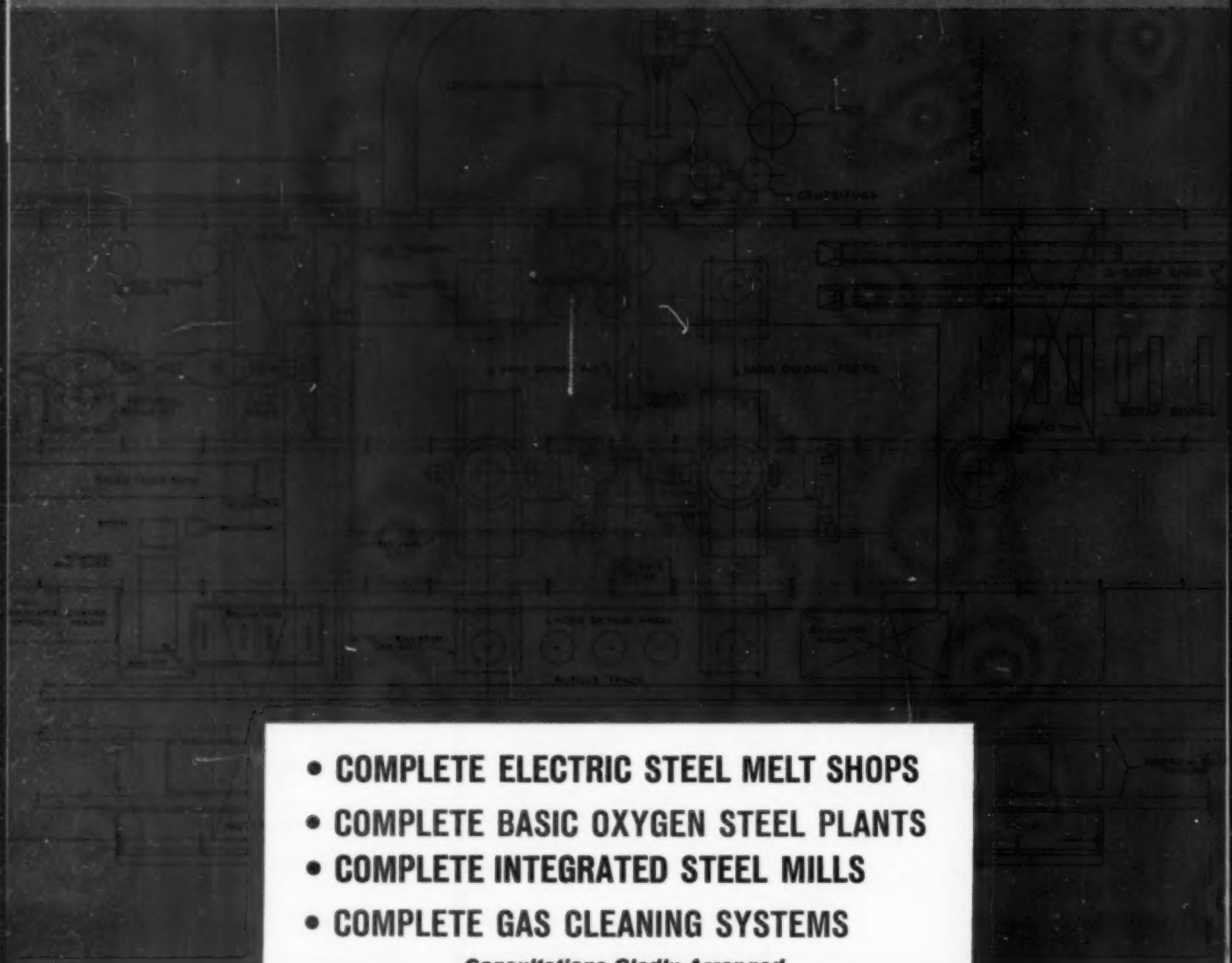

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PROCESS KINETICS OF OXYGEN STEELMAKING

The basic principles of chemical equilibrium and kinetics as they apply specifically to oxygen steelmaking are reviewed, the processes are analyzed, and some suggestions are made as to how oxygen steelmaking may be controlled more exactly.

by W. O. Philbrook

One of the most interesting features of steelmaking developments in recent years has been the way in which cross-fertilization of ideas and techniques, and the unifying influence of metallurgical oxygen have brought the processes closer together. Open-hearths are already approaching basic oxygen furnaces during refining, and the fuel torch is threatening to invade the arc furnace and basic oxygen domains. It may soon be redundant to have books, articles, or steelmakers specializing in one particular process. Although this paper is directed primarily toward basic oxygen steelmaking, it should be of some interest or value to steelmakers of all persuasions.

An attempt has been made to go beyond purely chemical aspects and to look also at any physical or mechanical features of the process that may limit its rate, efficiency, or controllability. As background for the process analysis, a brief review is given first of equilibrium limitations, chemical reaction mechanisms, and the dynamics of oxygen jetting. Space limitations have prevented a full development of these principles, and the reader who is especially interested in such aspects may wish to consult the more extended article and list of references that will appear in the *1961 Open Hearth Proceedings*. A complete explanation of the process is not yet possible, but an effort is made to point out the areas that need further study and suggest how to handle the problems.

Chemical principles

By logical arguments, based on equilibrium principles and the phase rule, it can be shown that the introduction of air or oxygen below the surface of hot metal through a submerged lance or tuyere can result only in an *acid* iron-manganese-silicate type of slag droplet and a gas bubble of prescribed $CO:CO_2$ ratio, both approaching equilibrium with the metal from which they were formed. Owing to imperfect mixing, the slag and gas of the real process are characteristic of metal more depleted in carbon, silicon, etc., than the average bath analysis at any moment.

Neither calcium nor CaO is soluble in liquid iron to any measureable extent. Consequently, any refining reactions that depend on the use of a basic slag can occur only at the slag-metal interface, never within the bulk of the metal. Every atom of phosphorus or sulfur to be removed must be brought to this interface at a time when slag composition and temperature are favorable for retaining them.

Equilibrium considerations lead to two important principles that must dominate any pneumatic steel refining process that depends exclusively on submerged lances or tuyeres: 1) oxygen injected below the surface of the metal *makes the metal* first, and the slag tends to be in equilibrium with the metal except for slowness in solution of any solid addition; and 2) the reactions within the metal are those characteristic of acid steelmaking, and basic refining reactions can take place only as metal is circulated past the slag-metal interface. The limited ability of the basic bessemer process to remove phosphorus except by an afterblow is an illustration of these principles.

Steel refining in the open hearth is uniquely different from the bessemer processes. The eternal characteristic of the open hearth is the driving flame over the slag, which is something like 10-million to 100-million times as oxidizing as the liquid steel beneath it. Equilibrium can never be attained here. The flame continually and inexorably oxidizes the slag, and the slag in turn oxidizes the metal. A steady-state is approached in which there is a strong oxygen gradient through the slag, with the lower layer of the slag perhaps 10 to 100 times more oxidizing than the main body of the metal. This may favor reactions that require oxidizing conditions, such as dephosphorization. In this sense, the process *makes the slag* first and the slag makes the metal. On the other hand, the open hearth is at a disadvantage when the time comes for deoxidation.

In the basic oxygen steelmaking processes, and to some extent in roof lancing, it is possible to

W. O. PHILBROOK is with the Metals Research Laboratory, Carnegie Institute of Technology, Pittsburgh, Pa. This paper was presented at the 1961 AIME National Open Hearth Steel Conference in Philadelphia.

manipulate the height of the jet and other factors to approach the effects of bottom blowing on one extreme or flame oxidation on the other. This is one important feature in the control of these processes.

Reaction Rates and Mechanisms

Chemical reaction rate theory leads to the expectation that the intrinsic rates of chemical reactions should be very fast at steelmaking temperatures. The real problem is the moving of material from one region to another. For practical purposes, this requires convective mixing or stirring because diffusion is too slow to be effective over distances of more than a few hundredths of an inch.

Most steel refining reactions take place at interfaces between slag and metal, metal and gas, or slag and gas, where reactants can get together or products escape. It is thought with good reason that there is a thin film called a *stagnant boundary layer* on each side of an interface, and atoms or ions have to get through these films by diffusion. Movement of fluids past the interface at high velocities will tear away part of the boundary layer and speed up the reactions by shortening the diffusion path, as well as by bringing up fresh reactants and whisking away the products so as to maintain steep concentration gradients. Stirring is probably the rate-controlling factor in most slag-metal reactions.

An interesting situation occurs in steel refining, where the most important cause of stirring is the carbon boil. The boil stimulates itself, both by its stirring action and by the large increase in interface areas, both gas-metal and slag-metal, that are created by the bubbles' rising and bursting through the slag.

Another possible bottleneck in reaction rates might be the nucleation of a new phase when a gas bubble or inclusion droplet is born. Bubble nucleation is probably the more difficult of the two, but even here there does not seem to be any serious long-range problem in steel refining. It is well known that CO bubbles originate at crevices on the hearth or side walls of a furnace, where tiny *seed* bubbles remain because the surface tension of the steel is too high for it to fill the smaller holes. There always seem to be enough of these nucleation sites to do the job, and the boil rate can be increased tremendously as needed to keep up with the rate of oxygen input, either because new sites come into play at slightly higher oxygen concentration levels or because the reaction just goes faster at the old ones. We shall return later to the point that bubble nucleation does not seem to place any serious limitation on the rate of carbon elimination once a good stirring motion has become established.

Oxygen jets and lances

In most top-blowing processes using jets or lances supplied with high-pressure oxygen, the flow rate is governed by the fact that the gas velocity in the throat of the nozzle can not exceed the velocity of sound. The maximum rate at which oxygen or air can be delivered from a frictionless nozzle operating with critical velocity in the throat is given by:

$$w_{max}\ (\text{lb per sec}) = 0.439\ \frac{p_1 D_0^2}{\sqrt{T_1}} \qquad [1]$$

where p_1 is the absolute pressure above the nozzle in psi, D_0 the throat diam in in., and T_1 the absolute temperature above the nozzle (°F + 460). Thus, for a given nozzle size, the mass flow rate of oxygen is directly proportional to the upstream absolute pressure and inversely proportional to the square root of the absolute temperature of the gas entering the nozzle. If the pressure and temperature are measured at the top of the lance or at the oxygen manifold, allowance has to be made for friction loss and temperature drop between the pressure tap and the nozzle. An accurate oxygen flowmeter compensated for both temperature and pressure is essential for precise control of the process.

The divergent section of a convergent-divergent nozzle is designed to complete the expansion of the gas from the critical pressure in the throat to the free discharge pressure, with maximum recovery of kinetic energy. The theoretical velocity of the emerging jet, its maximum kinetic energy, and its temperature, can be estimated from formulas for frictionless adiabatic expansion. For example, for a flow rate of 7000 std cfm through a 2.05-in. diam nozzle, the theoretical velocity of the oxygen leaving the tip of an ideal nozzle is about 1600 fps—a little over twice the velocity of sound in O_2 at the calculated temperature of −176°F. If the kinetic energy of this oxygen could be completely transferred to the bath, it would amount to over 700 hp available to sweep away the slag, penetrate the metal, and cause splashing and stirring. However, only a fraction of this is actually recovered as mechanical energy.

The kinetic energy of the jet is dissipated rapidly with distance from the nozzle because of interaction with the slow-moving molecules of the atmosphere into which the jet discharges. The supersonic core persists for a distance that depends on the Mach number.[2,3] For the example used, the core extends for about 16 throat diameters or 33 in. Beyond that distance, the jet is completely turbulent, and the velocity and pressure distribution across the stream follow bell-shaped curves as shown by Fig. 1. Velocity and pressure are plotted as fractions of the maximum value at the axis vs a relative radius expressed as a fraction or multiple of the radius at which the velocity is exactly half the maximum velocity. If absolute pressures and velocities were used, the curves would become wider and flatter

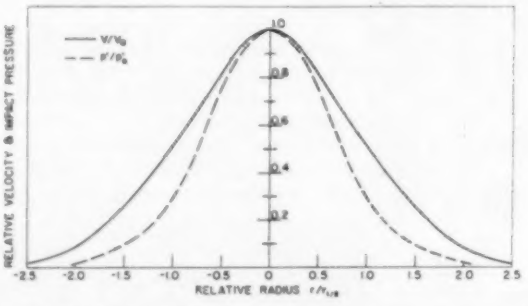

Fig. 1—Distribution of velocity and impact pressure across the diameter of a free jet. (After Anderson and Johns[2])

with increasing distance from the nozzle. The width of the curves out to negligible pressure falls within a cone of about 20° drawn from the nozzle tip.[4] The maximum impact pressure at the axis decreases inversely with (x/D_o) to the 2.3 to 2.5 power,[5] where x/D_o is the distance from the tip in nozzle diameters. Thus, the jet very rapidly becomes broader and softer in its action on the bath as lance height is increased.

The interaction of the jet with the atmosphere results in entrainment of some of the gas in the furnace, depending in amount upon lance height and pressure.[5] This does not make much difference to the over-all process if the gas is CO, but it can cause nitrogen pickup if air is present in the furnace. Loughrey[5] has reported the preventive remedy of charging 200 lb of carbo-coke to produce CO and purge air from the furnace before reblowing. The combined effects of the jet and the gases leaving the bath also result in some circulating currents[6] in the furnace space above the bath, bringing some of the gas into repeated contact with slag and metal droplets.

There is also some air drawn into the furnace opening, where it mixes and burns with the CO before leaving in the flue gas. In a detailed study carried out in a 3-ton experimental basic oxygen furnace, Maatsch, Plöckinger, and Wahlster[7, 8] found that about 3.3 pct of the total oxygen supplied to the process came from air. This atmospheric oxygen accounted for about half of the combustion of CO to produce the CO_2 found in the flue gases. This infiltrated air does not necessarily penetrate deeply enough into the furnace during the latter part of the blow to cause nitrogen pickup, but it does participate in the over-all heat and material balance. Since infiltration probably depends on furnace and hood design, condition of the mouth of the furnace, and lance height and pressure, and is therefore not readily predictable, it may be one of the exasperating problems in bringing the LD process under strict metallurgical control.

Interaction of the jet with metal and slag

The effect of an oxygen jet on molten metal and slag can not be observed with any fine detail within the furnace itself, nor can it be modeled correctly, so some degree of speculation is involved in attempting to describe the interaction.

The mechanical aspects of a non-consumable jet impinging on a liquid metal have been studied by Hammer, Kootz, and Sittard.[9, 10] If a vertical, high-velocity jet is directed upon the metal with the nozzle rather close to the surface, the jet punctures the surface and penetrates rather deeply to form a cavity of parabolic shape as depicted on the left in Fig. 2. The gas escapes along the walls of the cavity, and if its velocity is high enough, metal will be sheared from the walls and ejected as splashing droplets. The shearing forces also set up circulation currents below the surface and create a raised lip of metal around the cavity. Any slag on the surface may be rolled back behind the lip.

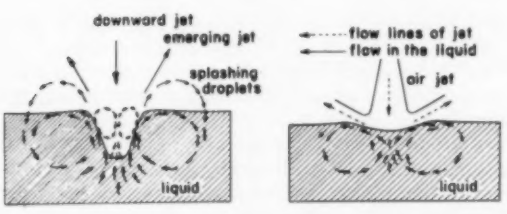

Fig. 2—Effect of an unconsumable jet on a metal. Left, low lance position with deep penetration and spashing. Right, high lance position with shallow penetration. (After Hammer, Kootz, and Sittard[9])

If the lance tip is raised or the jet velocity is decreased, the metal crater becomes broader and shallower and the splashing subsides, as indicated on the right in Fig. 2. The gases emerge at a much flatter angle to the surface. With increasing lance height, a point will be reached where the jet is no longer able to displace the slag, with the result that the slag layer may be lightly dimpled but direct contact between oxygen and metal will no longer be made.

A study of the absorption of a consumable gas from a jet by a liquid bath was carried out recently by E. G. Dubrawka[11] at Carnegie Institute of Technology. He blew CO_2 onto concentrated sodium hydroxide solution through a convergent-divergent nozzle at constant flow rate. His experimental conditions are by no means a faithful model of oxygen steelmaking, but the results correlate well with Kootz's observations[9, 10] and seem to be helpful in analyzing the process if the comparison is not pushed too far.

Dubrawka found clear evidence (see Fig. 3) of an optimum lance height for maximum CO_2 absorption, which correlated with his observations of the physical effect of the jet on the liquid surface. At the optimum height, the jet produced a deep and broad dimple in the bath surface and also a well-developed standing wave pattern spreading out from the dimple. As the nozzle tip was lowered from the optimum height, the depression became deeper and narrower, and splashing occurred as the tip was brought quite close to the bath; at the same time, the wave motion diminished. Physical conditions were similar to Fig. 2, left. On raising the nozzle above the optimum height, Dubrawka observed that the dimple became quite shallow, as suggested by Fig. 2 right, and the standing waves diminished to small amplitudes; efficiency of gas absorption again fell off. When a square or rectangular pan was substituted for a circular one of similar size, the standing waves failed to develop because of interference of reflections from the flat sides, and a considerable loss in efficiency of gas absorption was noted.

Further experiments at constant lance height showed that absorption efficiency increased with increasing bath diameter to a limiting value and then levelled off. Gas absorption also improved with increasing bath depth up to a limiting value, beyond which there was no further advantage. This critical bath depth was found to be the maximum depth to which stirring currents created by the jet could penetrate, and any further increase in depth resulted only in a stagnant layer in the bottom, as indicated by the flow patterns in Fig. 4.

Dubrawka tried to simulate the effect of a deep-seated carbon boil by bubbling nitrogen up through the bath from below while jetting CO_2 onto the surface. The additional stirring and surface disturb-

ance created by this artificial boil increased absorption efficiency by 40 pct, compared with otherwise identical conditions. Addition of a detergent to the bath to create a suds layer on the surface, somewhat analogous to a foamy slag, gave a further small increase in absorption.

Dubrawka's results showed that the absorption of CO_2 was favored by a broad and relatively deep dimple that presented a large surface for direct contact by the jet, but that absorption also continued as the gas swept over a considerable surface of the bath surrounding the jet and was enhanced by the additional surface presented by well-developed standing waves or a foamy layer. Increased stirring of the liquid, promoted by a deeper bath and particularly by an induced *boil*, was of great importance.

It does not necessarily follow that model experiments such as those described are quantitatively applicable to oxygen steelmaking or even indicate properly the stirring patterns near the point of impact or at depth. The absorption of oxygen by liquid steel is undoubtedly much more complete than in the models, but CO may be evolved close to the point of impact and may drastically modify the crater or wave pattern. Certainly, the intense heat generation in the zone of contact is not reproduced in the models. It is difficult to see how a quantitative description of these features of oxygen steelmaking can be obtained, so inferences drawn from imperfect models must be used with caution.

The basic oxygen processes

The background information that has been presented will now be applied in an attempt to develop a model or picture that will describe the refining of steel by oxygen processes and serve as a guide to better control of these methods of steel refining. It is logical to begin with the absorption of oxygen by the metal and then follow the reactions during the initial stages of refining and on through to the development of a strong carbon boil. Finally, it will be important to consider the reactions that are particularly dependent on refining by basic slags or are influenced by lance manipulation or other process variables.

Oxygen Absorption from the Jet

The first question to be answered, and a very important one, is: "What is the mechanism by which oxygen is received by the metal bath, and does this place any practical or theoretical limit on the rate at which oxygen steel refining can be carried out?" Several ideas of the prevailing mechanism of reaction of the jet with the metal have been proposed in the literature. In the writer's opinion it seems most likely that the first step in the reaction of gaseous oxygen with liquid steel must be something akin to chemisorption: oxygen molecules become attached to the metal surface, probably dissociating in the process. This step might be represented as

$$O_2 \text{ (gas)} \rightleftarrows 2\,O \text{ (at metal surface)}. \qquad [2]$$

The oxygen atoms at the surface may then diffuse inward as dissolved oxygen, $\underline{O}$, up to the limit of saturation of oxygen in the liquid iron alloy. Once the iron has become saturated, the adsorbed

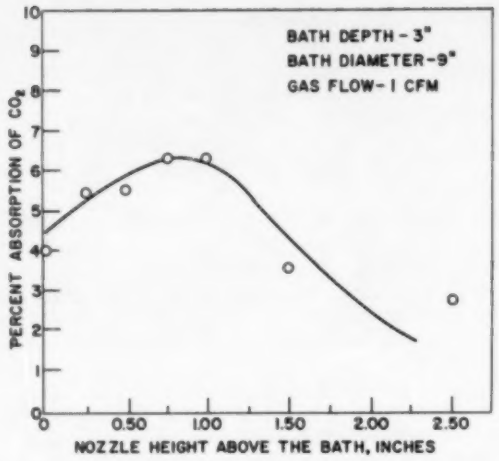

Fig. 3—Optimum lance height shown for absorption of carbon dioxide jetted onto a sodium hydroxide solution. (Dubrawka[11])

oxygen will react with iron atoms at or close to the surface to form liquid FeO. The FeO may be swept across the metal surface, to dissolve at some other point where the metal is not saturated with $\underline{O}$ or to join the slag. The dissolved oxygen will react with dissolved $\underline{Si}$, $\underline{Mn}$, $\underline{C}$, etc., as it finds itself in the company of atoms of these elements at suitable nucleation sites and in concentration levels where reaction is thermodynamically possible. There may even be virtually instantaneous reactions between impinging oxygen and dissolved elements until the metal surface has become depleted of these elements.

If the reagent were pure oxygen, there could be no diffusion transport problem in the gas phase, because the boundary layer film would simply be consumed continuously. In the case of a free jet that entrains gas from the atmosphere above the bath, a diffusion boundary layer may build up from the residual gas left behind as the oxygen is consumed. However, this film would be thin and continuously swept out of the crater of impact unless the jet were very *soft* because of a high lance or low pressure.

If the chemisorption hypothesis is correct, this first step does not seem to involve any significant bottleneck to the absorption of oxygen. The overall reactions at the surface are strongly exothermic. The heat of reaction far exceeds that necessary to raise the O_2 to reaction temperature, so the metal at the point of impact is strongly superheated and should readily supply the activation energy for adsorption. Owing to the mobility of the liquid metal and the shearing force of the oxygen stream, the metal surface should be renewed rapidly to provide an abundance of available sites for adsorption. The most adverse condition might occur near the start of the process before strong stirring currents have been developed in the metal. Here, the boundary layer problem for oxygen transport is reversed; that is, the surface film may be moved rapidly by the oxygen stream, but transport into the bulk metal phase may be limited by diffusion, with the result that the surface layer of the metal may quickly become saturated. In this event, liquid iron

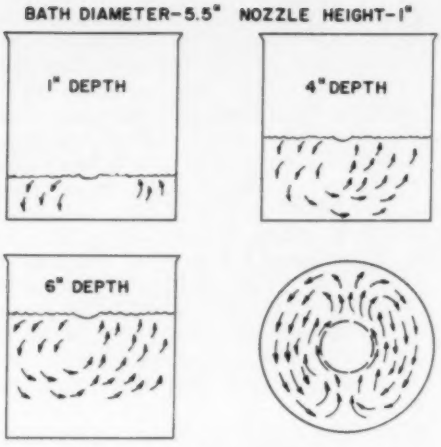

Fig. 4—Circulation patterns in bath where no improvement in absorption efficiency occurred beyond limiting depth of 4 in. Lower right sketch indicates currents in horizontal plane. (Dubrawka[13])

oxide would form at the interface, but this would be swept away continuously to expose new surface, if the lance position were not too high.

A very significant study by Larsen and Sordahl[12] showed that oxygen is absorbed very rapidly by a basic, oxidizing slag. Thus, any oxygen that escapes the primary crater of contact with the metal, but sweeps the slag at a low angle, should be captured. However, in the absence of a strong carbon boil or adequate slag volume, the slag may approach oxygen saturation.

Before checking these hypotheses against the evidence, it will be helpful to look at what might happen to dissolved O from the jet immediately after its absorption by the metal. There is no reason from either theory or observation to expect any significant delay in the reactions of O with Si or Mn to form inclusion globules as rapidly as reacting atoms are brought together by diffusion or mixing; so, the surface layer of metal would soon be depleted in manganese and silicon in the absence of convection. Carbon atoms could react to form CO at the gas-metal interface or diffuse into any bubbles dispersed below the surface by the force of the jet. However, the establishment of a general and extensive carbon boil will require the migration of O atoms to nucleating surfaces in sufficient concentration to provide the super-saturation necessary to cause bubble growth to begin on the *seed* nuclei in crevices. Some of the Si and Mn encountered along the path will have to be removed by reaction in order to build up super-saturation with respect to C. If the rate of migration is slow and the path is long, local regions of considerable supersaturation may develop temporarily.

There is experimental evidence that seems to fit the picture that has been developed. Plöckinger and Wahlster[9] have reported in detail their observations from heats blown in a 3-ton experimental basic oxygen furnace with continuous temperature measurement and frequent sampling of metal, slag, and flue-gases without interruption of the process. They found a relatively high level of CO_2 (about a 1:1 CO_2:CO ratio) in the flue gases during the first few minutes of the process. The proportion of CO increased as the rate of carbon drop increased, until the gas became practically pure CO by the time a linear rate of carbon drop had been established. Their oxygen balance[7] indicates that the amount of CO_2 in the gas was substantially higher than could be accounted for by air entrainment. This suggests that oxygen absorption by the metal was not quantitative during the first few minutes, and some of the jet oxygen reacted with CO in the furnace. Gas analyses reported from commercial operations have indicated a similar trend of high initial CO_2 content.

Plöckinger and Wahlster also reported a considerable super-saturation of the metal with oxygen, in comparison with both equilibrium and other steelmaking processes, which reached a maximum at about 2 to 3 pct carbon in the bath. Part of this excess oxygen may well have been present as suspended SiO_2 or MnO inclusions that had not yet joined the slag, and which were converted to Al_2O_3 in the aluminum-killed samples and included with the *total* oxygen. However, unreasonably high inclusion contents would be needed to account for all the *excess* oxygen. The oxygen super-saturation began to decrease at about the level of carbon content at which the maximum rate of carbon drop occurred.

These observations by Plöckinger and Wahlster indicate that there is some limitation to oxygen absorption, though probably not a serious one, during the first few minutes of the blow. This is probably the result of temporary saturation of the metal with oxygen in the region of jet impingement before bulk stirring of the metal has been established, rather than an indication of an inherent limit in the rate of oxygen absorption. The effect of lance height and manipulation on oxygen efficiency and initiation of the carbon boil during the first few minutes of the blow is a feature of oxygen steelmaking that deserves further study.

The Carbon Boil

From the analysis that has been made, the conclusion seems inescapable that the carbon boil is the dominating factor in oxygen steel refining just as it is in the open hearth. The vigor of its stirring action will be appreciated by anyone who has watched a steel ingot rimming at perhaps 1/50 to 1/100 the rate of carbon drop as in the basic oxygen process. Once a deep-seated boil has started, it becomes self-propagating from the combined effects of turbulent mixing to minimize concentration gradients and marked increases in the interface areas, including intermingling of slag and metal and the ejection of streamers of slag and droplets of metal into the gas phase[19]. Apparently a carbon boil can accelerate almost without limit to accommodate the elimination of oxygen as fast as it can be supplied. Larsen and Sordahl[12] obtained carbon-drop rates up to 7.6 pct per hr by flame oxidation alone in a small furnace fired with gas and oxygen, and Sims and Toy[18] reported carbon drops as high as 36 pct per hr in a small converter surface-blown with air from an inclined lance without deep penetration.

In a basic oxygen furnace blown at a typical rate of the order of 60 to 70 std cfm of O_2 per ton of charge, the bath produces a volume of CO equal to 2 to 2.5 times the volume of the metal bath at aver-

age temperature *each second*. There is no lack of motive power for stirring, and no scarcity of gas-metal interface throughout the volume of metal where the carbon-oxygen reaction can proceed. In fact, the bath may approximate a slurry of bubbles in metal, and the increased volume of slag and metal caused by their gas content must make the real stand-off distance of the jet considerably less than indicated by the nominal lance height. It is not surprising that over-all reaction rates should be high and oxygen absorption virtually quantitative under such turbulent conditions.

The writer is not aware of any evidence that a limit to the rate of oxygen absorption by a steel bath *on boil* has yet been attained experimentally, and it seems likely that the limit may be a practical one imposed by excessive splashing, slopping, or refractory erosion rather than one dictated by chemical features of the process. The only question of importance seems to be the mechanism for getting a boil started at depth in the oxygen process.

In the LD-type of vessel there are two stirring mechanisms for the initial transport of dissolved oxygen to the vessel walls and bottom or scrap surfaces where a deep-seated carbon boil might nucleate. One of these is the convection currents set up by the force of the jet as indicated by model studies such as those cited earlier. The other is natural convection resulting from differences in specific gravity between the newly-refined metal under the jet and the bulk of the hot metal, as proposed by Hauttmann[14]. In either case, some time would be required for the currents to spread in breadth and depth owing to the inertia of the metal, and this would account for a delay of a few minutes in the start of a deep boil. There is also a possible short-circuit path. If iron oxide formed under the jet or added as ore moves across the surface to the wall and dissolves in the metal there, a boil will start that will gradually work downward. Lance height, oxide additions, and furnace geometry are probably all involved in this delay period.

The carbon boil is likely to get under way more quickly in rotating furnaces such as the Kaldo or Rotor because the mechanical action will help to bring dissolved O to the walls. In the OLP or LD-AC process, lime particles suspended in the jet will be shot deeply into the metal because of their high momentum, and may serve as sites for CO nucleation at depth. Submerged lances may also disperse gas bubbles at greater depth than the jet. These mechanical variations may result in minor differences in oxygen efficiency and heat balance, but they are unimportant in relation to the over-all economy and control of the processes. The characteristics of a deep-seated carbon boil are over-riding and are essentially similar for all variations of oxygen steelmaking.

Slag-Metal Reactions

In the early discussion of equilibrium relations, it was stated that deep injection of oxygen will *make the metal* first by reactions characteristic of acid converting, and that basic slag refining can be accomplished only at the slag-metal interface. These conditions will prevail more or less strongly in basic oxygen steelmaking when a low to intermediate lance height is used, so that the oxygen jet sweeps away the slag and reacts directly with the metal. Only silicon, manganese, and carbon can be removed by the direct solution of oxygen into the metal. Basic refining and the approach to slag-metal equilibrium will be discussed first for low-lance conditions.

The extreme turbulence and high degree of mixing of slag and metal resulting from the violence of the carbon boil are conducive to rapid reaction and a close approach to equilibrium in oxygen steelmaking. The major adverse factor is the speed of these processes, which allows only a short time for shaping up a slag. This is offset partially by the high temperature level in the vicinity of the jet, but it is advantageous also to add mill scale, spar, or other fluxes and to charge the burnt lime in small pieces or inject it as powder with the jet in order to promote rapid formation of a reactive, basic slag.

In spite of the high rate of oxygen input, the dissolved oxygen content in the refined metal is closely related to the carbon content at normal tapping levels and falls within the same range as observed for the carbon-oxygen relation in the basic open-hearth[8, 15]. The FeO levels in the slag, as reported by numerous authors, generally tend to be the same as or lower than the average for open-hearth practice at equal carbon contents and slag basicities (see Fig. 1 of McBride,[15] for example). With deep jet penetration, one would expect lower slag oxidation than in the open-hearth, because the gas above the slag is CO rather than an oxidizing flame, and the constant passage through the slag of both CO and metal droplets from the bath will tend to hold the slag nearly in equilibrium with the metal. Manganese distribution approaches laboratory equilibrium values rather closely during the end of the blow under basic slag conditions, and according to Plöckinger and Wahlster,[8] the manganese goes to equilibrium almost immediately when the furnace is turned down. Again, one would expect a rapid and close approach to equilibrium during a terminal carbon boil after turn-down, because there is no sweeping flame to inexorably reoxidize the slag, as in the open hearth, and because relatively little *excess FeO* is stored in the slag if a penetrating jet has been used.

Some departure from equilibrium must be expected during the blow to provide the driving force necessary for the high rate of reaction. However, there is a rather consistent picture that the approach to instantaneous equilibrium between a matured slag and the metal is at least as good in the basic oxygen furnace as in other steel refining processes, and probably better than in the open hearth.

Phosphorus Elimination

The well-known requirements for extensive phosphorus removal are a basic, oxidizing slag and low temperatures. These conditions are best established by circumventing the normal tendency of the slag to work toward equilibrium with the carbon in the metal. It was pointed out earlier that the basic, bottom-blown basic bessemer process is not able to remove phosphorus effectively except by an afterblow[10], because the FeO content of the slag is *made from the metal* and lags carbon elimination. With top-blown processes, this situation may be reversed and made to parallel the open hearth more nearly, by working with a high, soft jet that will deliver much or all of the oxygen to and

through the slag rather than directly to the metal. The Kaldo process, for example, meets these conditions. It is necessary to establish an oxygen gradient that will keep the slag more highly oxidized than the metal to favor retention of phosphorus brought to the interface by convection.

Since phosphorus removal is favored by low temperature, it is advantageous to use scale and fluxes to aid in shaping up the slag quickly while bath temperatures are still low, and to hold down the finishing temperature. The powdered-lime processes are well suited for rapid development of a basic slag, but it seems doubtful that the physical immersion of lime below the surface has significant virtue for dephosphorization, because the temperature and oxygen level under the jet are not propitious for the lime to react with *P* at the point of injection.

The chief difficulty with any steelmaking trick that attempts to circumvent the normal striving of the process toward equilibrium is that the unbalance can not be held very long. Phosphorus is liable to revert from slag to metal as temperature rises or during the terminal boil after turn-down. This tendency can be minimized by holding the finishing temperature as low as teeming requirements permit and charging burnt lime just as the lance is raised to chill and thicken the slag. Another alternative is to blow down to a very low carbon endpoint, comparable with the afterblow in the basic bessemer process, and recarburize in the ladle while holding back the slag in the furnace. Owing to the tendency of the strong carbon boil in oxygen steelmaking processes to hold the slag FeO down toward equilibrium with carbon, the intrinsic ability of these processes to remove phosphorus is, at best, no better than the basic open hearth for equivalent slag volumes and basicities and carbon levels, and considerably worse if all the tricks are not used. This is illustrated by the comparison of residual phosphorus contents in Fig. 6 of McBride's paper[15] on the physical chemistry of oxygen steelmaking. If the phosphorus content of the charge is too high to be eliminated by a reasonable slag volume under equilibrium limitations, the safest procedure is to resort to a two-slag process. Three or four papers dealing with this subject may be found in the July 1960 JOURNAL OF METALS.

Sulfur Control

The sulfur input to the basic oxygen process is likely to be lower and under closer control than in the open hearth, because the former uses a small proportion of scrap that is usually of better quality. Furthermore, there is no sulfur pickup from fuel. Neither does there appear to be any significant loss of sulfur to the flue gases. Larsen and Sordahl[12] have pointed out that the effective oxygen pressure is not favorable for forming SO_2 from the surface of liquid pig iron or from a slag that is relatively deoxidized by a violent carbon boil. Consequently, nearly all the sulfur in the charge will be distributed between slag and metal at the end point.

The chemistry of desulfurization is the same in basic oxygen refining as in other basic steelmaking processes and has been discussed recently by McBride.[15] Desulfurization is favored by high slag basicity, high slag volume, high temperature, and low FeO. The basic oxygen process may have a slight advantage over average open hearth practice on all of these counts, and also in closer approach to equilibrium. McBride[15] found that sulfur distribution ratios for basic oxygen processes scattered about the equilibrium line predicted from laboratory studies, with a number of values of (pct S)/[pct S] running up to 12 to 14 compared with a maximum of 8 to 10 in the open hearth. Assuming that there is no carryover of blast-furnace slag into the oxygen furnace and that the burnt lime is reasonably low in sulfur, a slag volume of 220 to 250 lb per ton at a lime-silica ratio of 3.0 or higher should remove 50 to 60 pct of the sulfur in the metallic charge. This compares favorably with a 35 to 50 pct sulfur removal in the open hearth. There is no reason to expect any significant differences in desulfurizing ability among the several variations of oxygen steel refining.

Other Elements

The behavior of other alloying elements in oxygen steelmaking is predictable from their known physical chemistry and behavior in other steelmaking processes. The soluble elements more noble than iron, such as copper, nickel, cobalt, molybdenum, and tin, will remain in the steel. Chromium will exhibit the same type of behavior as it does under oxygen lancing in electric furnaces, and owing to the high temperatures obtainable in the basic oxygen process, it is possible to blow down to very low carbon contents without excessive chromium loss, provided that proper attention is given to slag volume and composition. The potentialities of the oxygen processes for making alloy steels have not yet been fully explored, but such considerations are beyond the scope of this paper.

Process control

It is the aim of forwarding-looking oxygen steelmakers to bring the process under close enough control so that it can be satisfactorily described by mathematical models. Computers can then be used to calculate the charge proportions and blowing times that will result in the desired end-point analyses and temperature within acceptable tolerances. Once this has been accomplished, the next steps to more or less complete automation of charging and blowing become essentially matters of economics and the design of hardware. A first step toward computer control has already been accomplished by Slatoskv.[18] and further efforts along these lines are undoubtedly going on in many places.

Pneumatic steelmaking processes are probably more amenable to automation than either open-hearth or electric furnaces. The converter processes are basically simpler in regard both to materials handling and to the number of process variables involved, but the strongest advantage is that the heat balances and chemical reactions are almost quantitatively related to the oxygen supply.

Oxygen and Heat Balances

These two subjects are inextricably woven together in the oxygen process, and an accurate oxygen balance is essential to the mathematical prediction and control of the process. The heat balance, and hence the control of tapping temperature, is very sensitive to small variations in process chemistry. Generally speaking, the accuracy of weighing, sampling, and analyzing raw materials, measuring hot metal temperature, and metering oxygen

flow, all require much stricter standards than have been customary in steel mill practice if satisfactory control is to be established.

The foregoing discussion has pointed out that there is some air infiltration during the process, and that a portion of the jet oxygen escapes absorption by the bath and burns some CO to CO_2 within the furnace during the first few minutes. Both factors are probably related to lance height, and air infiltration will also depend on furnace and hood design and condition of the lining. Later in the process, a high lance can be used to increase slag oxidation in order to aid phosphorus removal, whereas a low lance will give minimum slag FeO. Slatosky[16] has incorporated a correction for lance height in his model. All of these variations from theoretical efficiency of oxygen usage will normally be reflected in the amount of heat that is attributed to burning carbon to CO or CO_2. This will introduce errors in the calculation of scrap requirement or finishing temperature, and probably in the end-point carbon as well. There does not seem to be a way of predicting these variations quantitatively by scientific principles or model studies. They will have to be established by experiment. Unfortunately, the normal errors in describing the process variables are so great now in many cases that it is not even possible from operating data to demonstrate the effect with assurance, much less discriminate well enough to establish correlations.

It is regrettable that there are still some gaps in the fundamental thermodynamic data needed to describe the process. A revision of the writer's calculations of three years ago[17] to bring them into agreement with more recently published thermal data[15, 19] raised the calculated scrap consumption by about 1.5 pct. This is still low compared with experience at Aliquippa[6, 18] and Fontana[1] because no allowance was made for combustion of CO or CO_2. Information in the literature on the heat content of hot metal and slag and on some of the heats of reaction are not very reliable, and additional research is needed here and on some of the equilibrium and activity data. In the meantime, it is possible to get by with empirical factors if consistent material balances can be obtained.

Kinetic vs Equilibrium Models

The various thermochemical descriptions of oxygen steelmaking that have been published thus far, including Slatosky's[16] computer model and a number of heat balances, have considered only the over-all process from charge to products without regard for the details in between. One might ask whether any advantage might be gained from a kinetic model that would attempt to predict the course of the process, and whether such a model is feasible. In my opinion, an attempt to write a mathematical model for the first half to two-thirds of the blow would serve no practical purpose and have little likelihood of scientific success. A continuous or kinetic model for the latter part of the process would help in making heats to high-carbon specifications by *catching on the way down* and perhaps for making corrective additions if the initial charge balance were not exactly correct.

As a suggestion, a kinetic model might be devised to enter the process after the injection of a specified quantity of oxygen, calculated to bring the carbon in the range between 1 and 2 pct. A correction determined from experience would be needed to adjust for air infiltration and CO_2 formation. It would be assumed that silicon and manganese have been eliminated and lime dissolved and that a near approach to slag-metal equilibrium has been established. An over-all heat and material balance up to this point would predict the momentary temperature and analyses. A step-wise model for successive small increments of oxygen might be devised to reproduce the course of the blow from this time to the desired end point. It would be necessary to incorporate an experimentally determined correction for lance height and degree of departure from equilibrium. A heat balance must run concurrently with the material balance to provide continual adjustment of temperature, and allowance for external heat losses must be built into the model. The job is not an easy one, and some compromises and approximations will no doubt be necessary to keep the model simple enough for reasonable calculation time on a computer that is not too elaborate for practical purposes. A computer model of the process that is good enough to have real practical value does not seem unattainable.

Summary

The basic principles of chemical equilibrium and kinetics have been reviewed as they apply specifically to oxygen steelmaking, and the characteristics of high velocity jets impinging on liquid metal have been summarized. Oxygen steelmaking processes have then been analyzed to learn which of these process variables is limiting at successive stages of the blow and what variations in control may be possible. Finally, some suggestions have been made on the steps by which oxygen steelmaking might be made subject to more exact control with the aid of computers.

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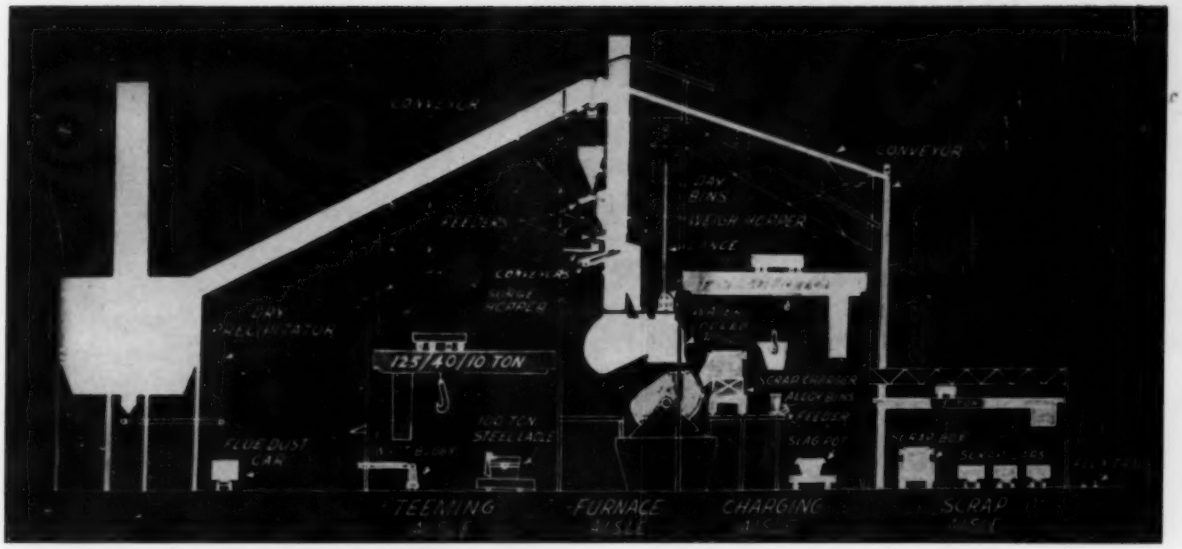

BASIC OXYGEN STEELMAKING IN COLORADO

A description of the first basic oxygen steel plant in the Rocky Mountain area, this article also includes operating data since start-up on the 11th of July.

by George Grosvenor

As early as 1955, CF&I considered the basic oxygen furnace process of manufacturing steel as a means of creating additional capacity for the Pueblo, Colo., plant. At that time, however, the process had not progressed to the point of meeting the grades of steel necessary to satisfy customers' needs. The result was the building of an additional open-hearth furnace in the existing 16-furnace shop.

Since completion of the new open-hearth furnace, great strides have been made with the basic oxygen process. Two very important improvements that affect CF&I are: the ability to use additional amounts of scrap in the charge, and the capability of tapping heats in a wide range of carbon (0.05 to 1.00 pct) contents. In 1959 the decision to build a basic oxygen furnace was made; it was to have an ingot capacity of 50,000 net tons per month. Planning was started immediately for the engineering and construction of such a shop, and ground breaking took place on May 3, 1960.

At the same time, experiments were undertaken to increase by approximately 15 pct the output of hot metal from the plant's four blast furnaces. This would mean producing a total of 110,000 net tons of hot metal per month. The additional iron was required to replace some of the iron the open hearth must give up to the basic oxygen furnace shop. Means adopted included redesigning the blast furnaces to take a carbon lining, increasing the number of tuyeres from 12 to 16, maximizing stove capacity, utilizing higher grade ores, and injecting natural gas and other materials into the blast furnace.

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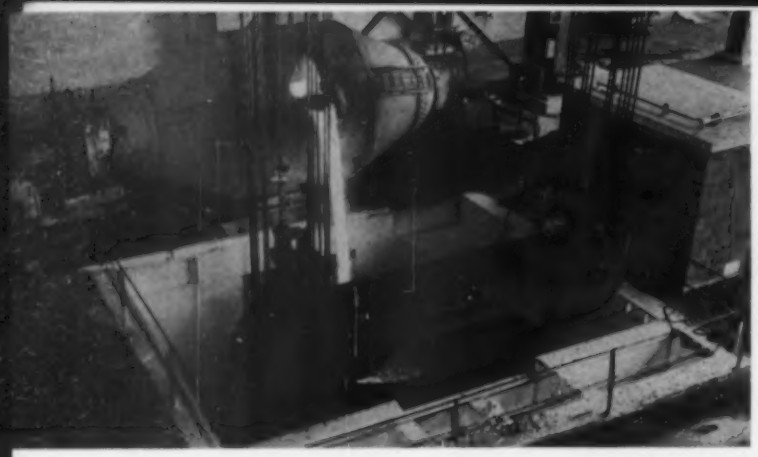
The basic oxygen furnace

The basic oxygen shop is a two-furnace plant with one furnace operating and the other being relined or held on a stand-by basis. Size of the furnace shell is 18.5 ft ID and 27 ft high with a volume of 2810 cu ft after lining. The outer shell is made of 2-in. thick steel plate of welded construction. Nominal heat size is 100 net tons with a rated capacity of 100 tons per hour. The plant is being broken in on 80 ton heats, but after optimum operating conditions have been worked out, heat size will be increased to 100 tons and at a later date to perhaps as much as 125 tons. Lining is of tar-bonded magnesite brick with lining life expected to be between 125 and 150 heats with the initial one-shift-per-day operation of the plant. Operating on a three shift per day basis should give a considerably greater lining life.



Arrival of Hot Metal

Charging Hot Metal

The furnace utilizes a water-cooled, copper-tipped lance of BAMAG design. It is lowered through the furnace hood into the vessel itself through controls in the pulpit. Oxygen at 175 psi passes through the 2.05-in. diam orifice at the tip of the lance. Fluxes and other materials, such as burnt lime, spar, mill scale, and ore, are charged into the furnace through the flux chute during the blow. All of these additions are automatically controlled from the pulpit.

Dry-type electrostatic precipitators of Buell design are employed to remove solids from the flue gas, and a pug mill is to be used to remove dust from the precipitators hoppers. Oxygen furnace fumes first pass through a water-cooled hood into a spray chamber where water sprays clear the fumes of heavier particles. The fan capacity is rated at 300,000 cfm at 350°F. A 700 hp motor drives the fan, creating the vacuum of 10-in. of water at the precipitator header.

Charging and teeming facilities

Scrap charging is accomplished with a Calderon charger which is capable of charging the furnace in 1 min with approximately 22 tons of light scrap (45 lb/cu ft) from one 1200-cu-ft charging box. Each of these boxes is of sufficient size to carry the scrap requirements of one heat. When operations call for a charge of 30 pct scrap or more, it is necessary to fill a charging box with a denser scrap. With relatively high cost and limited quantities of hot metal, it is expected that the inital 20 to 25 pct scrap charge will be increased to 25 to 30 pct and more.

The CF&I basic oxygen plant is the first in which the Calderon scrap charging system has been used. The particular machine has been designed to meet the specific requirements of CF&I plant, but the principle remains the same as for previous installations with several angles of repose used in the charging box to provide an even flow of scrap and with the scrap passing through a 90° angle so as to provide a parallel flow of scrap pieces.

Hot metal is brought into the basic oxygen shop in 125-ton torpedo-type ladles. The metal is automatically weighed and poured into 90-ton transfer ladles for charging into the basic oxygen furnace. Analysis of the hot metal varies little from day to day due to an elaborate ore bedding system and the lack of variation in the ores used. Average analysis is 4.20 pct C, 1.15 to 1.35 pct Si, 0.45 pct Mn, 0.030 pct S, and 0.3 pct max P. Temperature of the metal is measured by optical pyrometer during transfer to the furnace ladle; it is normally about 2500°F.

In designing the plant care was taken to provide against any bottle-neck in the teaming aisle; thus the teeming table area is capable of handling four 125-ton heats. Top pouring practice is followed for both killed and rimming steels with exothermic hot tops used for special grades. Ingots are teemed in 11,000 lb 23x24-in. and the 7200 lb 18x18 sizes.

Other facilities

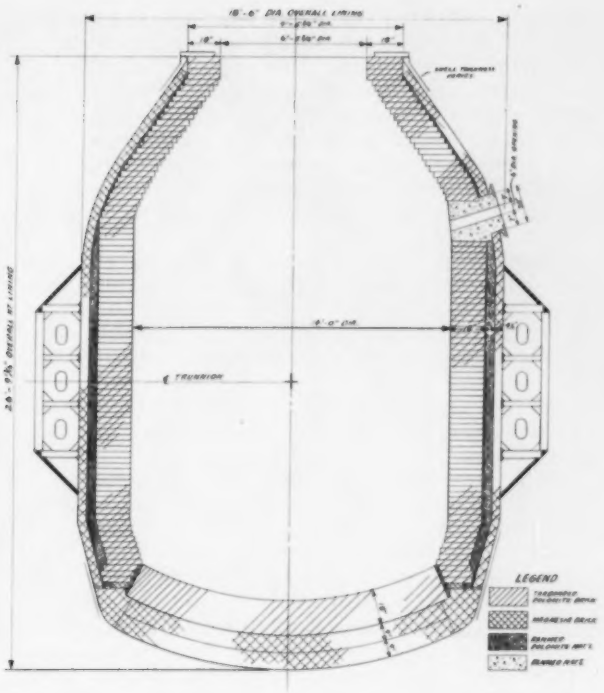
Dimensions of the building are as follows: scrap aisle crane, runway 65 ft wide by 100 ft long; charging aisle 65 ft wide by 400 ft long; furnace aisle 40 ft wide by 500 ft long; and teeming aisle 65 ft wide by 500 ft long. Total height of the building is 166 ft.

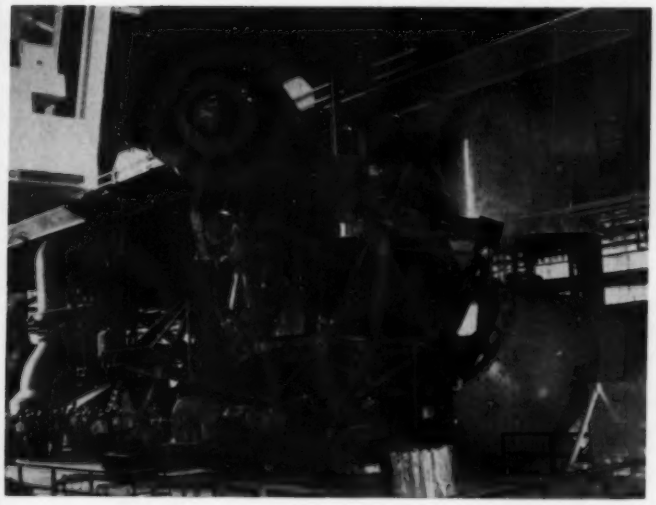
Equipment consists of one 15-ton crane for scrap handling; one 125-ton crane in the charging aisle; and one 125-ton crane in the teeming aisle. Three 7000 lb fork trucks with rotary heads are used to handle alloys and refractories. Car pullers are used to move blast furnace torpedo cars. A rubber-tired car mover is used to move scrap and flux cars. A 8-ton elevator, located south of the furnaces, handles spare parts, refractories, and lift trucks to the various levels. An enclosed rubber belt conveyor system handles fluxes from the dumping station to the day bins and then to the furnace hoppers.

CF&I's

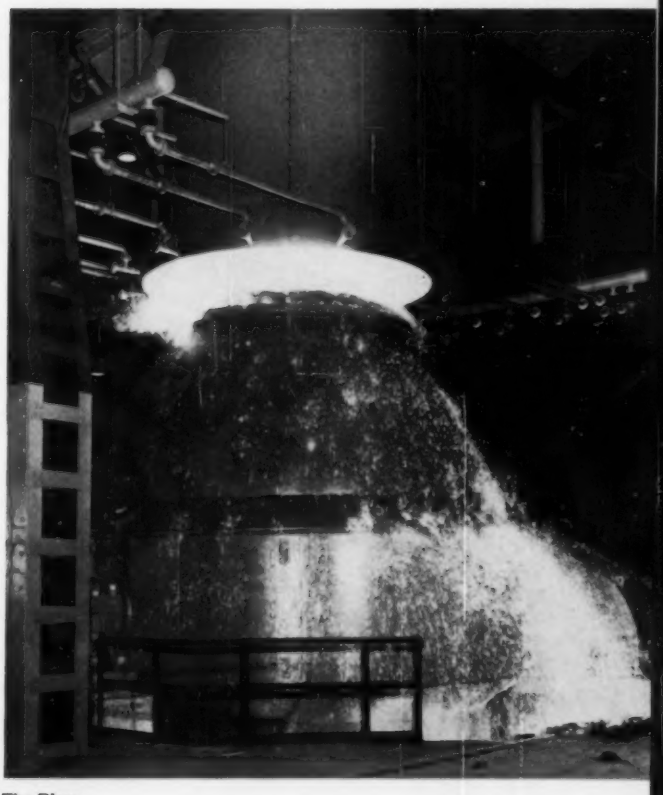
Basic Oxygen Steel Plant

Pueblo, Colorado



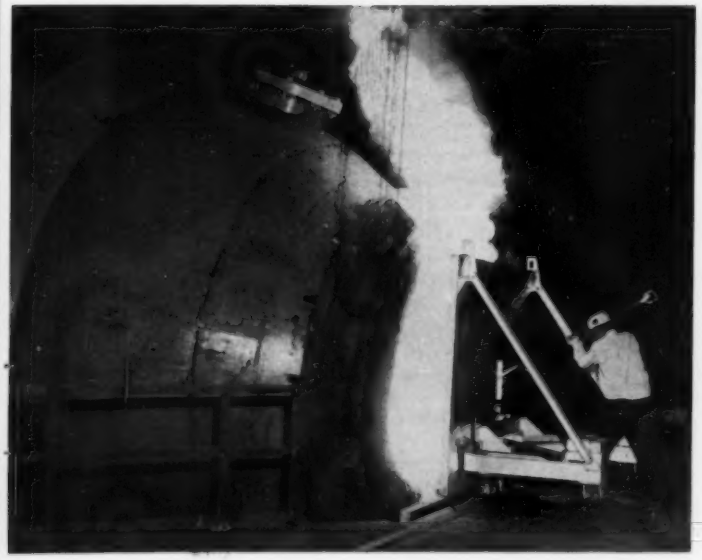


Scrap Charging



The Blow

End of Blow



Transfer cars for the teeming ladle and slag pot are capable of working in tandem using power from only one of them.

Initially, eight 80-ton teeming ladles and 20 slag pots of 260 cu ft capacity are available for use in the shop. Present slag handling methods used in the open hearth have been incorporated in the new shop. Ladle repair and relining are done in the central repair shop which also services the open hearth.

Water supply consists of a combination of primary and recirculated water, received under 80 psi. For lance cooling at the rate of 250 gal per min, water pressure is raised to 150 psi. Hood cooling water is used at 80 psi and then re-pumped for spray water at 50 psi.

The normal complement of controls and indicators are incorporated for water temperatures and pressures, oxygen pressure, lance clamping, flue gas temperature, automatic flux batching system, hot metal and bath temperatures, and steel sampling. All controls, including those for the precipitators, are located at the basic oxygen plant pulpit.

Belt conveyors lift burnt lime, spar, mill scale, and ore from the flux unloading hopper to the five 4000-cu-ft day bins and a batch hopper with a capacity of 480 cu ft. The hot metal station can handle two torpedo cars at a time, and the scrap aisle trackage can handle 30 cars, including six cars under the crane. A standard gauge track into the south end of the building is used to unload refractories, alloys, and spare parts onto the dock and storage area.

Combined service operations for both the open hearth and basic oxygen furnace shop include ladle relining, stopper making, mold preparation, hot-top preparation, narrow gauge transportation, ingot stripper and storage yard, scrap preparation, and necessary clerical help for ordering supplies and materials.

The oxygen plant, built and operated by Linde, is located outside the plant and furnishes 280 net tons of oxygen per day with a line pressure of 185 psi. Storage facilities provide for liquid equivalent of 10,000,000 cu ft of oxygen and a gaseous surge of 200,000 cu ft.

Operations

A little over a year after the ground breaking—on Tuesday the 11th of July 1961—the first heat was tapped from one of the two basic oxygen furnaces. The charge for that first heat of 80 tons of steel consisted of 20 tons of scrap and 70 tons of hot metal.

Present plans anticipate 100,000 net tons of steel to be produced in the open hearth and 50,000 net tons of steel to be produced at the basic oxygen furnace shop each month. Products of the Pueblo plant of CF&I range from low carbon rimmed steel (0.05 pct max) for wire, through the intermediate carbon grades (30 to 50 pct) for seamless tube steel to high carbon (0.60 to 1.00 pct) for rails, fastening, and grader blades. Inasmuch as scheduling of the two blooming mills requires the full range of steel continually, both the open hearth and basic oxygen furnace shop will be required to produce them.

During the first month of operation of CF&I's basic oxygen shop, 80-ton heats have been produced on a one-shift-per-day basis using charges made up of 25-pct scrap and 75-pct hot metal. Ore and mill scale have been used as cooling agents in the final phase of the blow, and metal recovery has averaged 85.59 pct (total Fe in hot metal, plus scrap, plus cooling ore/Fe in ingots). Oxygen at 175 psi has been blown at the rate of 6500 cfm, and lance height has been held at 88 to 90 in. above the slag metal interface.

On a tap-to-tap basis the time sequence has been as follows:

Charging time:	
Scrap	45 sec
Hot metal	1 min 15 sec
Oxygen ignition	45 sec
Blowing time	23 min
Turn down & reblow	6 min
Sampling & tapping	15 min
Total time, approx	47 min

Tapping temperatures, measured by an immersion pyrometer have been in the range of 2870° to 2900°F, depending upon the grade of steel. During the first month, steels ranging from 0.04 to 0.70 pct C have been produced without difficulty. Below 0.25 pct C, ladle recarburization has been practiced, but above that level it has proved possible to catch the carbon without undue difficulty.

Further plans call for changing over to full round-the-clock operation with one furnace on blow producing 100 ton heats. This will call for increased oxygen flow rates so as to maintain a blowing time of approximately 20 min. In addition, a larger diameter nozzle will be substituted for the present 2.05 in. diam nozzle when increased flows are necessary.

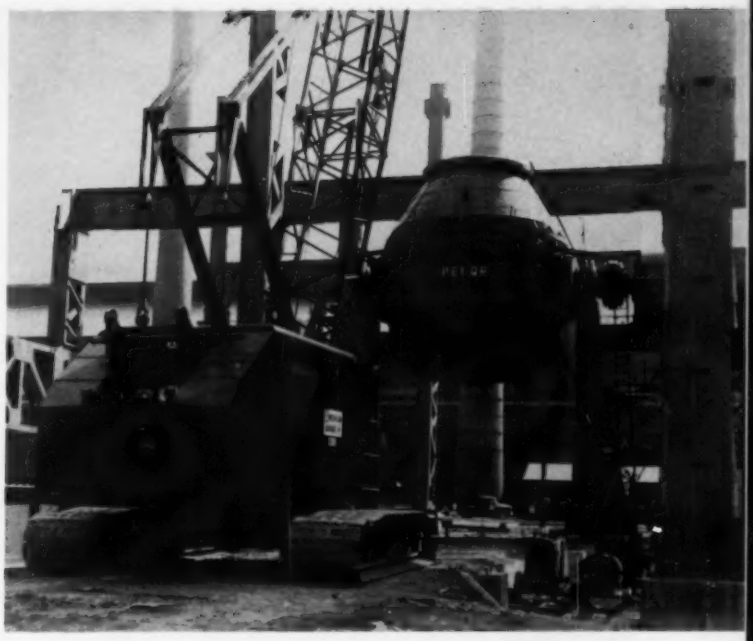
As the plant moves into full-scale operation at a later date, heat size may be extended to 125 tons with resultant operating rates considerably in excess of the rated 100 tons per hour. With high hot metal costs and relatively abundant scrap, it is anticipated that experimental work with an oxygen-fuel lance will be undertaken so as to make possible a 45-pct scrap charge.

But these matters are something for a future article. All that we can now say definitely is that we have completed more than one month of successful operation of our basic oxygen plant. Results, which have been fully up to expectations, are shown in the following table:

Operational Data of the CF&I Basic Oxygen Plant

	First Week July	First Week Aug. (Third Week of Opn)
Heats, no.	58	34
Heat size, tons	72.64	77.82
Operating hr	104	40
Output, tons	4279.10	2645.85
Operating rate, tph	41.13	66.15
Tap to tap, min	59	56
Charge to tap, min	55	51
Blowing time, min	23	22
Hot metal charge, pct	76.87	79.17
Iron analyses, avg		
P, pct	0.269	0.276
S, pct	0.030	0.043
Mn, pct	0.27	0.26
Steel analyses, avg		
P, pct	0.017	0.018
S, pct	0.017	0.018
Mn, pct	0.63	0.58

CLEVELAND RECEIVES BASIC OXYGEN STEELMAKING



Courtesy Koppers Co.

Marking Jones & Laughlin's second venture into basic oxygen steelmaking, start-up of the Cleveland plant will put in operation what will, for several months, be the largest basic oxygen furnaces in the world.

by J. A. Glasgow

It was November 1959 when workmen began clearing ground at the Cleveland works of Jones & Laughlin Steel Corp.; this marked the inauguration of a modernization program which included the building of a basic oxygen furnace shop with two 200-ton vessels. The two furnaces are expected to produce at approximately 200 tph and will replace eight 175-ton open-hearth furnaces constructed from 1924 through 1930 when the Cleveland works was owned by Otis Steel Co. The decision to install the basic oxygen furnaces was based on operating experience with two similar-type furnaces at the Aliquippa works of Jones & Laughlin Steel Corp.

Now nearing completion, the new plant will include a scrap yard, charging aisle, furnace aisle, teeming aisle, precipitators, and other installations which are described in the following sections.

Scrap yard

The scrap yard, located adjacent to the charging aisle, is approximately 96 ft wide and 250 ft long. Two 15-ton, single-hook magnet cranes will unload scrap from gondola cars into 2400-cu ft charging boxes. The yard will be serviced by six, standard gauge railroad tracks which will accommodate 25 cars of scrap. Charging boxes will be moved, two at a shift, by an engine.

J. A. GLASGOW is open-hearth superintendent, Cleveland works, Jones & Laughlin Steel Corp., Cleveland, Ohio. The original paper was presented at the 1961 AIME National Open Hearth Steel Conference in Philadelphia.

Charging aisle

The charging aisle is 67.5 ft wide and 500 ft long. Two identical 300-ton main hoist cranes, which will service the furnaces with scrap and hot metal, will also handle slag pots and move iron and steel ladles for repairs.

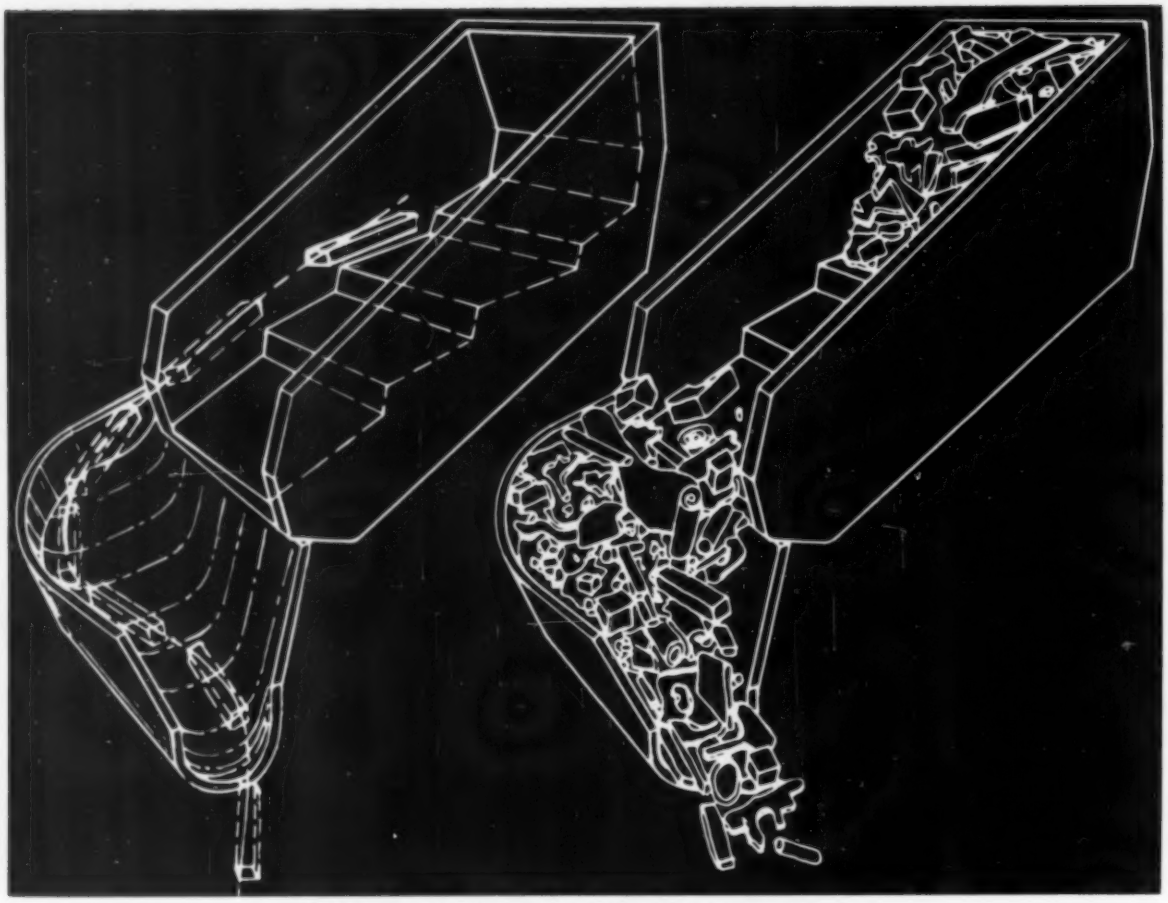
Hot metal will be delivered in submarine-type ladles to the south end of the charging aisle where it will be weighed on a track scale, and poured into 230-ton hot-metal transfer ladles for delivery to the furnace. Scrap will be delivered at the north end in charging boxes which will be picked up by crane, placed on a Calderon charging machine where the necessary weight adjustments will be made, and then charged into the furnace. The scrap charging operation can be completed in approximately 1 min.

Furnace slag will be poured into two 500-cu-ft slag pots carried on a self-propelled slag car that will run under the furnace; these slag pots will be placed onto self-dumping cars for delivery to the slag dump.

Iron and steel ladle skulling, relining, and drying will be performed at the north end of the charging aisle where two ladle repair stations are located.

Furnace aisle

The furnace aisle—38 ft wide, 500 ft long, and 160 ft high—consists of four floors above the pit



Calderon scrap charging system applied to the basic oxygen furnace.

level: the charging floor, service floor, larry car floor, and the bin floor. A 15-ton elevator services all floors.

At the pit level, transfer car tracks for the slag pot cars and steel ladle cars are equipped with splash shields to direct falling debris away from the running tracks. Both the slag pot and the steel ladle cars are powered by individual electric motors which receive power through a collector rail arrangement.

The concentric-type basic oxygen furnaces are constructed of 2 to 2.5-in. thick steel plates in two pieces: the nose cone and the barrel which includes the bottom. Outside measurements are 21 ft, 8.5 in. in diam, and 27 ft, 10.5 in. in length. The nose or mouth of the furnace is made to allow an opening from 6 to 8 ft in diam when newly lined. Lining of the new furnaces will follow the procedures currently used at the Aliquippa works. (See p. 631 of this issue, JOURNAL OF METALS).

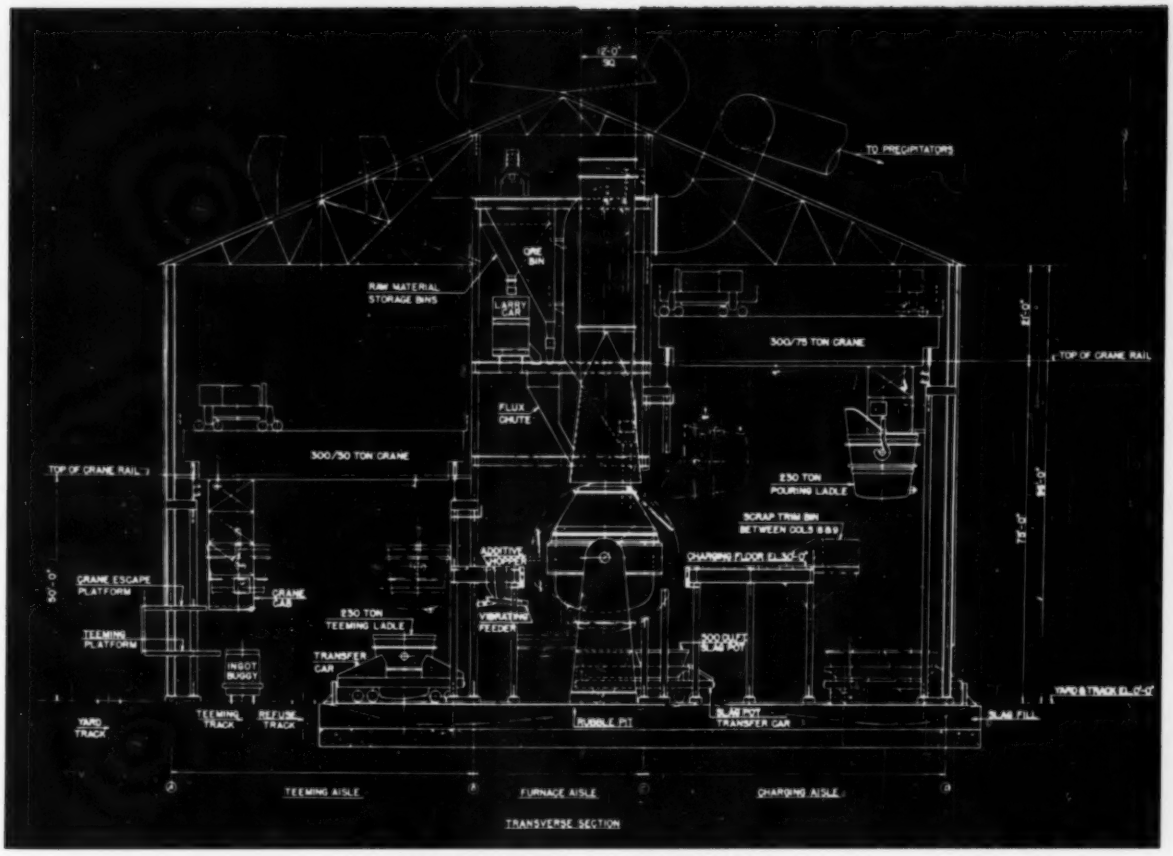
Trunions are fitted with Torrington spherical roller bearings driven by two 200-hp motors through a 50-to-1 worm gear and a 4-to-1 helical gear. Either motor is capable of rotating the furnace under normal operating conditions. Furnace rotating speed is controlled by a five-point, reversing, adjustable controller which allows a maximum speed of 1.56 rpm.

On the charging floor, the operators pulpit is located 8.5 ft from the furnace in order to provide better vision for furnace operation. Auxiliary controls for furnace movements for charging, tapping, slagging, and making alloy additions are provided on the tapping and charging sides of the furnace. Since the furnaces are 100 ft apart and the drives are located to the outside, the operating floor between the two furnaces is clear.

The fume hoods over the furnace are equipped with three banks of water sprays. These will control the temperatures of the gas before it passes into a refractory-lined spark box. Solids will be deposited on a grating for removal on the service floor level. Material carried away by the cooling water will flow to retention tanks suspended under the charging floor, and the cooled gases will be drawn through a duct to the precipitators. A section of the stack immediately above the entrance to the spark box is situated on rollers to allow service to the hood by a 10-ton auxiliary crane. This crane services hoods, oxygen lances, and furnace rebuilds; it can be used on all service floors.

The oxygen lances are 42 1/3 ft long, 8 in. in diam, copper tipped, and water cooled. A system which permits two lances to be in operating readiness on each furnace will be used. Changeover from one lance to another can be accomplished very quickly; the system eliminated the necessity for clamping and positioning the lance at various heights during blowing. Hoist speeds on these units vary from 46 to 90 ft per min.

Oxygen will be delivered from a plant of Air Products, Inc. located adjacent to the property. Two 250-ton units will supply oxygen at 250 psi and allow blowing at flow rates up to 20,000 cfm.



J&L's basic oxygen plant at Cleveland, Ohio.

Storage space for 750,000 std cu ft of gaseous and 750 tons of liquid oxygen will be available.

Raw materials delivered to the shop will be transferred to the storage bins by conveyor belt. These storage bins will have the following total capacities: 360 tons of burnt lime, 75 tons of fluorspar, 110 tons of mill scale, 250 tons of ore, and 30 tons of coke. Ore bins with automatic weight-control scales are provided at each furnace to allow ore additions to be made as desired. Four storage bins containing 175 tons of alloys are available for ladle additions.

Teeming aisle

The teeming aisle—67.5 ft wide, 700 ft long, and 57 ft high—will be serviced by two identical 300/50-ton cranes. There are two teeming platforms, each 270 ft long with a track cross-over between them and entry tracks at opposite ends of each platform. A sufficient number of molds for four heats will be in the pits at all times. An auxiliary track is provided for the slag pot and rubble cars between the teeming tracks.

Precipitators

Furnace gases will be cleaned in electrostatic precipitators to 0.05 grains per std cu ft of gas. This is well below any existing air pollution code. The precipitron unit—one of the largest of its kind—is designed not only to handle the gases from the new furnaces, but also the gases from the three oxygen-equipped, open-hearth furnaces currently in use. The unit consists of eight individual electrostatic precipitators with separate controls which will allow for the operation of any desired number of units. Four 325,000-cfm fans, powered by 1250-hp synchronous motors will pull the gases through the duct system. The precipitant will be passed through a pug mill and trucked to the sinter plant.

Other features

An electronic computer—similar to the one currently in use at the Aliquippa works—will be used by the furnace operator to calculate the proper proportions of hot metal, scrap, and fluxes to be charged into the furnace. This will help to insure that the various grades of steel are tapped at the desired temperatures.

Water cooling for the furnace hoods, oxygen lances, and service water are all separate systems. Hood and lance systems will use treated water.

Outlook

The new shop will be capable of producing a heat of steel in approximately 1 hr as compared to the 4 to 5 hr required with electric furnaces and the 6 hr for oxygen-equipped open-hearth furnaces for a heat of the same tonnage. It is anticipated that the steel produced in the new furnaces will be used in the production of hot- and cold-rolled sheet.

Construction is nearly completed, and the furnaces are expected to go into production sometime this month.

GREAT LAKES' BASIC OXYGEN FURNACES— World's Largest!

by R. B. Morgan

This article describes the design features of the 300-ton basic oxygen steel furnaces being constructed at Great Lakes Steel Corp.'s Detroit plant. Start up of the 1¾ million tons per year plant is scheduled for 1962.

As part of a current expansion and improvement program of National Steel Corp., two, 300-ton basic oxygen furnaces are under construction at Great Lakes Steel Corp.'s Detroit plant. The overall program will increase the corporation's product mix, thereby reducing the costly cyclical fluctuations caused by the seasonal demand of the automotive industry for steel products.

The economic savings realized from lower capital expenditures and the expected reduction in operating costs justified the change to the basic oxygen steelmaking process. The resultant decision to build unprecedented size furnaces was influenced by the necessity to utilize the existing 300-ton teeming facilities and the need to produce large ingots—up to 35 tons. In addition, data extrapolated from contemporary basic oxygen furnaces producing 100-ton heats, indicated the feasibility of operating larger vessels.

General plant layout

The basic oxygen plant will cover an area approximately 280 x 810 ft. A site, west of the no. 2 open-hearth shop, was selected for construction of the new plant in order to utilize the existing bessemer steel works buliding for scrap loading, and the no. 2 open-hearth shop's teeming aisle. The basic oxygen shop has been designed to permit the installation of a third vessel at a future date.

The new plant will have a three-aisle arrangement consisting of charging, furnace, and teeming aisles. Scrap will be handled inside the bessemer building and transferred to the furnace aisle; teeming will be performed on the present no. 2 open-hearth shop pit which will be expanded.

Scrap handling and charging

Incoming scrap will be delivered in gondola cars to the scrap handling area located on the west end of the charging aisle. Three railroad tracks in that area will accommodate 25 gondola cars. The 1500-cu-ft scrap charging boxes will be placed between the two sets of tracks for loading. A majority of the scrap will be loaded with the existing 15-ton crane; the balance will be loaded by the 45-ton hoist on the 150-ton crane.

The furnaces will be charged by a self-propelled charging car travelling in front of the furnace on a wide-gauge track on the operating floor. Scrap adjustment will be made west of the no. 1 furnace, and the cars will carry two boxes for each normal 30-pct scrap charge. Each box will have its own tilting mechanism, and scrap will flow directly from the tilted box into the furnace.

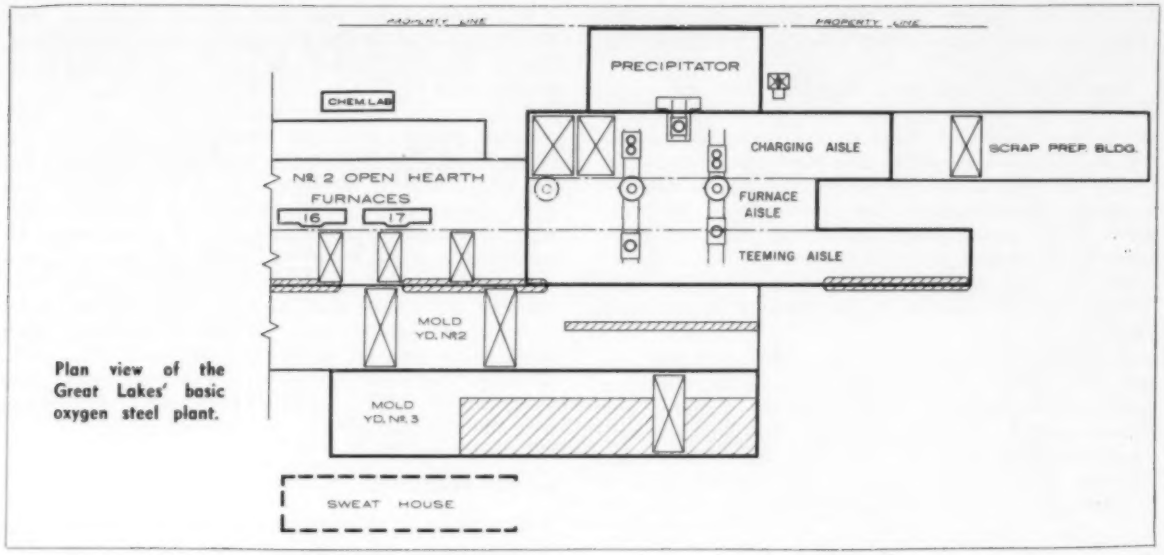
Hot metal from the Blast Furnace div. at Zug Island will be transported in 110- and 200-ton submarine ladle cars. The hot metal will be reladled adjacent to the charging aisle near the furnaces. Two pouring stations will be provided, one being for emergency use. A 275-ton ladle with an extended lip will be moved on a self-propelled transfer car to the furnace where a 350-ton ladle will pour the metal into the furnace. The metal charge weight will be secured by differential weighing from track scales located under the submarine cars.

Fluxes delivered to the track hopper will be transported by conveyors into overhead storage bins. Eight bulk storage bins will provide the following storage capacities: 960 tons of burnt lime, 300 tons of sinter or pellets, 250 tons of coke breeze, and 200 tons of spar. Gravimetric feeders, located under each of the eight bins, will batch the preselected amount to the weighing hoppers over each furnace. These feeders can be remotely controlled from the pulpit floor. The flux charge will be directly fed from each weighing hopper into the furnace through a 24-in. water-cooled chute. Flow rates from the weighing hoppers can be regulated by varying the gate valve position by pneumatic control from the pulpit.

Furnace

The concentric-top furnaces are designed for a nominal capacity of 250 tons, but are being built to carry 300 tons. The furnace configuration was achieved by empirical extrapolitation from lines of the successfully-operating basic oxygen furnaces and from test model studies. The more spherical shape of these larger furnaces is a departure from the contemporary vessels which has been designed

R. B. MORGAN is assistant superintendent, no. 2 open hearth shop, Great Lakes Steel Corp., Detroit, Mich. This paper was presented at the 1961 AIME National Open Hearth Steel Conference in Philadelphia.



Plan view of the Great Lakes' basic oxygen steel plant.

to accommodate expected oxygen blowing rates up to 30,000 cfm.

The 2.5-in. thick, dished-bottom shell without peep holes will be supported by a separate trunnion ring and equipped with a four-piece sectional replaceable cone lip ring. Each furnace will have dual drives, with two separate motors mounted on opposite sides of the furnace. All four motors on each furnace will be attached to the bull gear drive. Variable-voltage electrical controls with one motor-generator set for each vessel will rotate the furnace at 1.4 rpm. Either motor-generator set will power either furnace in an emergency, and the rotation speed can be maintained with one of the four motors out of service.

The initial 9-in. refractory linings will be burnt magnesite brick, serving as tank linings next to the steel shell. A 4-in. rammed layer will be used between the tank and the working lining of the barrel and nose sections. The working lining of the barrel will consist of 24-in. magnesite tar-bonded brick. The nose area working lining will be 21-in. magnesite tar-bonded brick, and one 18-in. course of tar-bonded magnesite brick and 18-in. burnt magnesite brick will make up the lining of the bottom.

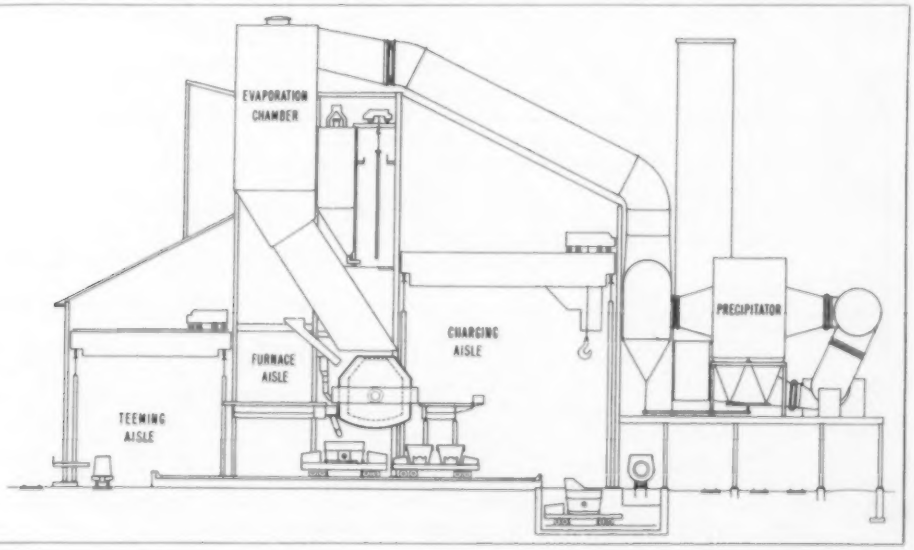
The four principal ladle additives will be stored in bulk bins loaded by fork lifts that have been elevated to the service floor via the service elevator. These four bins will discharge into a motor-operated larry car. The larry car will run to a feed hopper and vibrating system. The miscellaneous alloys with lower demand requirements will be stored in bins on the operating floor and will be fed into the feed hopper and vibrating system. Each furnace will be provided with duplicate facilities.

Teeming and ingot handling

Steel will be tapped into 300-ton teeming ladles. The ladles will be carried into the teeming aisle by a self-propelled transfer car.

The three existing 300/40-ton pit cranes will service the teeming aisle. The present teeming aisle will be extended 470 ft, and a fourth 220-ft pouring platform added.

Ladle relining will be performed in the two existing ladle holes at the extreme east end of the pit.



Cross-section of the plant.

Nozzles will be set from the outside. A new 70-units-per-turn stopper rod oven will replace the present rod room.

The existing mold yard, together with the present 50- and 25-ton cranes, will be used to clean, spray, and build up drags of big-end-down, open-top and bottle-top molds. The big-end-up, hot-top molds and heads will be worked in a new hot-top yard located adjacent to the north side of the mold yard. The hot-top yard will be an elevated platform arrangement and will be serviced by a 75-ton crane to strip and set heads. A mold sweat shed will be erected parallel to the north of the hot-top yard.

Slag will be collected in two, 480-cu ft capacity pots (the same slag pots currently used in the open hearths) mounted on self-propelled transfer car. The transfer car running under the furnace will carry the pots into the charging aisle. Either the 350- or 150-ton crane will transfer the slag pots to a truck and trailer unit for delivery to the dump.

Gas and fume control

The gas and fume control system will be a dry electrostatic type, consisting of the following main components: hoods, evaporation chambers, duct work, headers, precipitators, fans, and stack. The precipitators, inlet and outlet headers, fans and stack will be set 30 ft above ground level over the hot-metal and flux receiving stations.

A steam, boiler-type hood, about 19-ft in diam and 54-ft long located directly above each furnace, will conduct the gases into the evaporation chamber. Gases within the hood will be cooled and humidified by the direct application of water near the upper portion of the hood.

Each furnace will have a 33-ft diam by 67-ft high evaporation chamber above the hood. Steam will be injected into the chamber to condition the gases. Each chamber will have two emergency by-pass dampers.

The gases from the evaporation chambers will be carried over to the precipitators or inlet header. Heavy dust particles that fall out in the inlet header will be collected in the bunker hopper.

Three dry-type electrostatic precipitators will consist of two units, each containing four electrical fields operating in series to yield 24 ft of treatment zone. There will be eight discharge electrodes in each electrical field. The electrodes will be automatically cleaned by a vibrator, and dust will be collected in bunker hoppers under the precipitators.

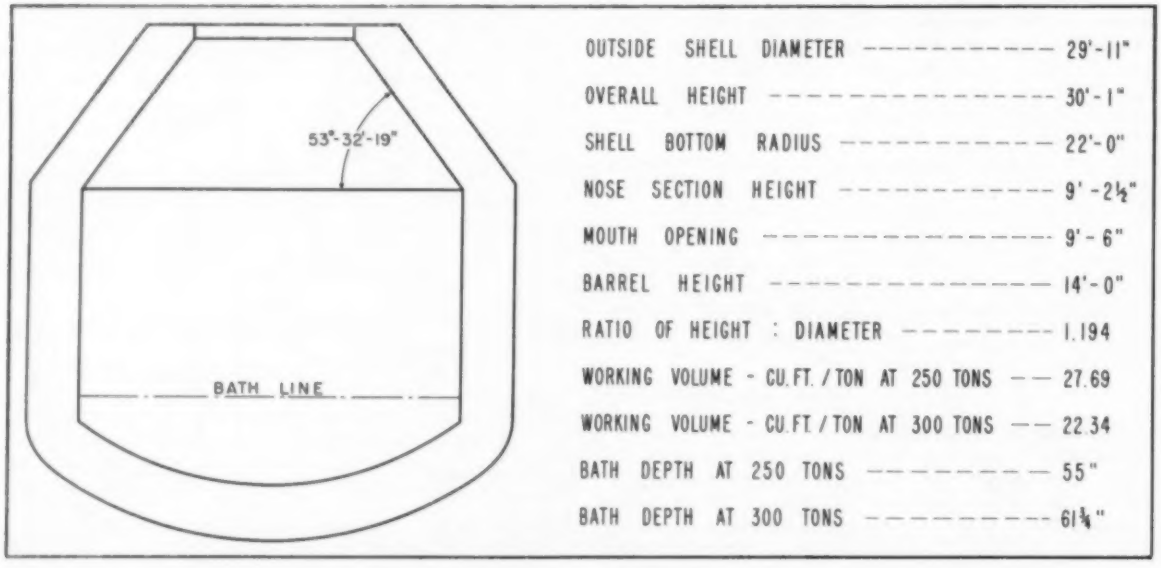
A 19.5-ft diam precipitator outlet header will receive the gases and deliver them to the draft fans. Three 600,000 cfm (at 500°F) induced draft fans, with one on stand-by, will pull the gases through the cleaning system. The fans will force the gases into ducts leading to a stack 17 ft in diam and 200 ft high.

Other features

The present 500-ton oxygen plant will be supplemented by an additional new 350-ton unit. The oxygen will be produced at 165 psi and recompressed to 275 to 600 psi for transmission to the basic furnace site. There will be 600,000 cu ft at 275 psi available in storage near the furnaces. Twenty million cu ft liquid storage capacity will be provided at the oxygen producing units.

The lances will be capable of handling up to 30,000 cfm oxygen. Their length will be approximately 48 ft, including the head, which will be equipped with four hose connections: two for oxygen, one for water supply, and one for return water. A computer will be provided for the primary function of calculating charge materials to improve end-point temperature control, and log process data. Provisions have been made for future expansion of the computer for later incorporation of close-looped control.

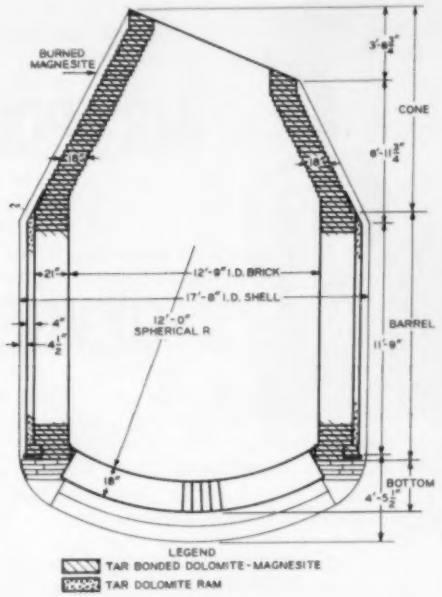
Construction of the plant is now underway, and Great Lakes' 1¾ ton per year plant will go on stream during the course of 1962.


53°-32'-19"
BATH LINE
OUTSIDE SHELL DIAMETER ---------- 29'-11"
OVERALL HEIGHT ---------- 30'-1"
SHELL BOTTOM RADIUS ---------- 22'-0"
NOSE SECTION HEIGHT ---------- 9'-2½"
MOUTH OPENING ---------- 9'-6"
BARREL HEIGHT ---------- 14'-0"
RATIO OF HEIGHT : DIAMETER ---------- 1.194
WORKING VOLUME - CU.FT./TON AT 250 TONS -- 27.69
WORKING VOLUME - CU.FT/TON AT 300 TONS -- 22.34
BATH DEPTH AT 250 TONS ---------- 55"
BATH DEPTH AT 300 TONS ---------- 61¾"

Cross-section through the furnace.

BASIC OXYGEN FURNACE LININGS AT ALIQUIPPA

Basic oxygen steelmakers have been searching for refractories which will provide lining life at reasonable cost. This article is a progress report on the results of using several types of basic brick at the Aliquippa works of J & L.



Based on a paper by J. N. Albaugh

Since the introduction of the LD basic oxygen steelmaking process to the North American continent in 1954, steelmakers have been investigating refractories with the aim of finding a brick which would be the least expensive per ingot ton, and provide a sufficiently long lining life.

Initial tests at Aliquippa works were run on tar-bonded magnesia brick containing 70 to 74 pct MgO and 10 to 15 pct CaO. The first eight linings averaged 131 heats with refractory consumption of 51 lb per net ton for 54-ton heats with oxygen flow of 3800 cfm. Lining failures on the charging side of the furnace led to the installation of a pilaster 27-in. thick on that side of the furnace; the result was an increase in lining life; however, with improvements in charging techniques, the pilaster has been eliminated. It was found that failures did not occur below the bath level in the furnace; consequently, the brick work in the lower 6 ft of the furnace was reduced in thickness from 27 in. to 22.5 in., and subsequently to 18 in. without any detrimental effects. This reduced the working lining by 12.5 tons and proved to be economical.

In March 1958, heat-size was changed from 54 to 81 tons, and attempts were made to reduce refining time by increasing the oxygen flow rates. Rates of 6500 cfm and above increased refining rates from 2.5 to 4 tons per min, but resulted in greater wear in the cone section. To alleviate this problem, the working lining of the cone section was increased from 13 to 16 in.

Changing over to tar-bonded dolomite-magnesite brick, lining life averaged 190 and 159 heats with brick consumption at 19.65 and 24.10 lb per ingot ton, respectively, for two investigating periods. Further improvement in lining life was obtained through the use of 100 pct tar-bonded magnesite brick. The initial six linings had an average life of 233 heats with brick consumption 12.28 lb per ingot ton, and a campaign time of 7½ days. To further increase the lining life with the magnesite refractory, 21- and 24-in. bricks were installed in the barrel section of the furnace to replace the 18 in. brick. As a result, the following 21 linings averaged 259 heats with a brick consumption of 16.77 lb per ingot ton. However, full utility of the 24 in. brick was not realized because of consistent lining failures in the 16-in. cone section. For this reason, the use of 24-in. brick has been temporarily discontinued, and 21-in. bricks are being used exclusively in the barrel section. As a result of these changes, lining failures are again confined to the cone section, and evaluations are underway with the use of 18-in. brick in that area.

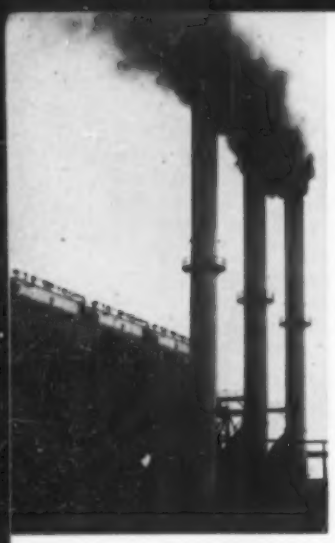
The method of operation also influences lining life. In the fourth quarter of 1960, production varied from 13 to 18 turns per week with an 8 hr down time between turns. This method of operating seriously affected lining life. In the third quarter, we operated at 20 turns per week in producing 30.88 heats per day in 11 campaigns which averaged 258 heats per lining and used 17.17 lb of brick per ingot ton. In the fourth quarter, we averaged 20.31 heats per day in eight campaigns which averaged 218 heats per lining and used 20.40 lb of brick per ingot ton.

The working lining contour is currently 18-in. thick in the inverted bottom, 21-in. in the barrel, and 18 in. in the cone section. The weight of this working lining is approximately 168 tons. The permanent tank lining of burned magnesite brick remains the same as originally installed. At present, the lining in service contains 60 to 62 pct MgO, 32 to 34 pct CaO, and the balance auxiliary oxides of silica, alumina, and iron oxide.

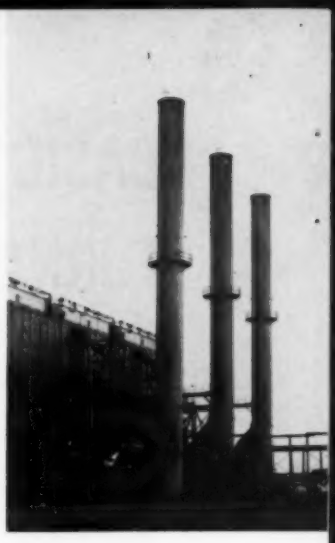
This type of brick had an average lining life of 240 heats for 1960, and three linings in January provided lining lives of 240, 247, and 251 heats, for an average lining life of 246 heats.

We currently have three sources capable of supplying 100-pct magnesite tar-bonded brick and will receive a trial supply of tar dolomite brick from a fourth producer. Since we are very much interested in cost, we are hopeful of obtaining a tar dolomite brick which will provide satisfactory service for an 8 to 10 day run at maximum tonnage output.

J. N. ALBAUGH is with the Aliquippa works of Jones & Laughlin Steel Corp., Aliquippa, Pa. The original paper was presented at the 1961 AIME National Open Hearth Steel Committee meeting in Philadelphia.



AIR POLLUTION CONTROL IN OXYGEN STEELMAKING



Successful installation of electrostatic precipitators on large open-hearth furnaces led to their use for the LD basic oxygen steelmaking process. This article describes the air pollution control system and presents results of tests and operation at Kaiser Steel's Fontana, Calif., plant.

Based on an article by J. H. Smith

When the management of Kaiser Steel Corp. decided to build and operate an oxygen steelmaking facility, it also made the decision to underwrite considerable new air pollution research and to install several million dollars worth of air pollution control equipment.

Historically, the company has always been cognizant of the air pollution problem and its particular importance to southern California. Kaiser Steel's Fontana plant is located in San Bernardino County, some 45 miles east of Los Angeles; it is nestled in the center of a horseshoe of mountains. Due to this geography and proximity to the Los Angeles smog problem, the original plans for the steel mill, made in 1942, stipulated that all known practicable air pollution controls were to be incorporated. This policy has been followed in all succeeding expansion programs.

Kaiser personnel, who were handed the problem of controlling air pollution from the new facility, were faced with a unique challenge. The LD basic oxygen steelmaking process was new, and the facility was the world's largest at the time of its design. Air pollution control of such magnitude had never before been accomplished.

To complicate matters, the selection of air pollution control devices to do the job was limited not only by the chronic water shortage in southern California, but also by problems created in the disposal of waste water. The water system at Fontana is closed, and the mill uses 1400 to 1600 gal of water per ton of steel as compared to the national average of 65,000 gal per ton.

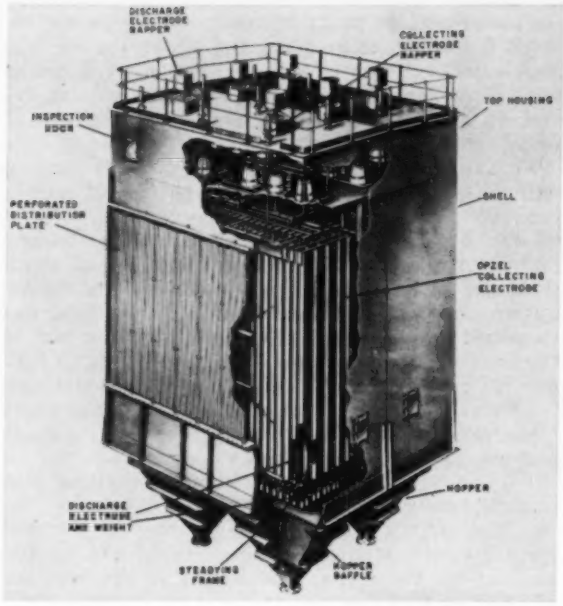
In the event that a wet cleaning system were to have been incorporated in the basic oxygen steelmaking facilities, those responsible for the design would also have to incorporate water pollution controls into the same system. And since the State Regional Water Pollution Control Boards have set water quality standards demanding water treatment of most effluent (thus further increasing water consumption), we directed our thinking in terms of dry collection devices, such as cyclones, baghouses, and electrostatic precipitators.

Kaiser Steel had been instrumental in pioneering the first successful installation of electrostatic precipitation on large open-hearth furnaces using a hot-metal charge; therefore, when the basic oxygen steel plant controls were being considered, it was only natural to look first to electrostatic precipitation.

Electrostatic precipitation

Electrostatic precipitation had not been tested on basic oxygen steel plants at the time Kaiser personnel were faced with the task of developing a satisfactory dust cleaning system for the proposed installation. Instead it was necessary to rely on theory and adaptation of past experiences with the open-hearth electrostatic precipitators.

J. H. SMITH is director, Air Control and Research, Kaiser Steel Corp., Fontana, Calif. This paper was presented at the 1961 AIME National Open Hearth Steel Conference in Philadelphia.



Typical electrical precipitator with three electrical sections similar to the six-section unit installed by Research-Cottrell on Kaiser's basic oxygen furnaces at Fontana, Calif.

Utilizing this information, design specifications for three precipitators for the oxygen furnaces were established. Each precipitator was to be 53 ft 3.25 in. long, 52 ft 2.75 in. wide, and 56 ft 2.75 in. in height, measuring from the bottom of the hoppers to the top of the insulator compartment. Each precipitator was to be divided into two sections, and each section was to have six energized compartments. The discharge electrodes were to be 25 ft 6 in. long, while collecting plates were to be 6 ft long and 23 ft 6 in. in height.

Each of the three precipitators was designed to handle waste gases having a peak rate of 202,000 std cfm or 405,000 cfm at designed operating conditions. Temperatures in the precipitators were estimated to be 560° ± 30°F. Pressure, to be —10 in. water gauge. The gas analysis calculated for maximum blow conditions was 5 pct CO_2, 14 pct O_2, 61 pct N_2, and 20 pct H_2O.

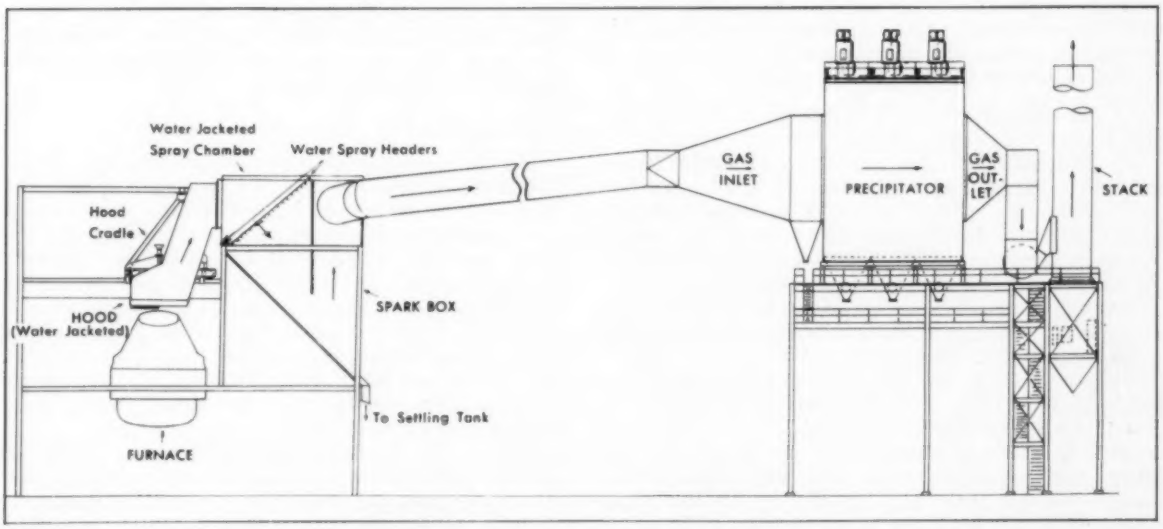
Dust samples obtained from other installations led us to believe that 5 to 15 pct of the dust would be larger than 1.0 micron, 45 to 65 pct would be between 0.5 and 1.0 micron, and 20 to 50 pct would be smaller than 0.5 micron. Analyses of the dust indicated 93 pct to be Fe_2O_3 with the balance mainly MnO and small quantities of CaO, SiO_2, and alkalis. It was further calculated that there would be about 4400 lb of dust per 22 min heat with a peak evolution of 347 lb per min during the blow. The estimated maximum dust concentration was 6.0 grains per cu ft at inlet conditions.

Relying further on experience with the open-hearth precipitators, perforated plates were proposed and gas velocities not exceeding 3 fps were specified to insure good performance. Conical hoppers were also specified.

Previous experience has proven that good distribution through the precipitator is essential if good efficiencies are to be achieved. We believe that the maximum deviation from mean velocity should not exceed ± 10 pct. Dust build-up had been encountered with the conventional hopper design. Modifications to the open-hearth precipitators indicated conical hoppers should prevent the dust from hanging up and causing arcing to the discharge electrodes.

Design requirements

Furnace hood design was a critical problem. The various hoods being used in other basic oxygen steelmaking shops were studied before establishing design criteria for the hood to be incorporated in the Fontana installation. The hood was designed for water cooling and was made in sections for easy access when replacement becomes necessary. The sections consist of a number of vertical water-tight channels with an inlet connection at the bottom of each channel and an outlet at the top. The water to the hood is supplied by a circular header. Connec-



Oxygen furnace gas cleaning system designed by Kaiser Engineers at Jones & Laughlin's Aliquippa plant; it is similar in design to the Fontana unit. Courtesy, Research Cottrell.

tions from this header are made by hoses to each vertical channel in the bottom sections. The discharges of the bottom sections are connected to the inlet of the section above so that each supply of water follows a vertical channel to the open discharge bosh at the top of the hood. It is possible to determine that each channel is properly cooled by inspection of the free-water discharge in the bosh. These channels were designed for a water velocity of 6 to 6.5 fps to prevent deposition of solids from the quality of water to be supplied.

Performance requirements stated that the vendor should guarantee the following for normal operating conditions:

1) That the suspended solids in the exit gas leaving the outlet flue system of the installation would not exceed 30 lb per hr for any 1 hr including the rapping cycle.
2) That except for a period or periods aggregating not more than 3 min in any 1 hr would the discharge from the flue system be,
   a) As dark or darker in shade as that designated as No. 2 on the Ringelman Chart as published by the U.S. Bureau of Mines, or
   b) Of such opacity as to obscure an observer's view to a degree equal to or greater than does smoke described in subsection (a) above.

Performance tests

From November 5, 1958 to May 8, 1959 performance tests were made on the BOSP fume-control system. During this period, the size of the heats (in terms of metal charged) was gradually increased from 80 to 131 tons. The percentage of hot metal in the charge varied from about 72 to 87 pct.

These tests were conducted jointly by Kaiser Engineers, Kaiser Steel utilities dept., and Kaiser Steel air control and research dept. personnel. The tests indicated satisfactory performance of the basic oxygen steelmaking plant fume-control system. The variables measured included gas flow; pct CO_2 at the furnace mouth and at the precipitator inlets; pct CO at the precipitator inlet; pct water vapor at the the precipitator inlet; dust loadings; precipitator efficiency; static pressure along the system; velocity distribution between the precipitator units; and temperatures at the furnace mouth, at the precipitator inlet and outlet, and in the stack.

The results of the test data indicated:

1) No explosion hazard in the fume system under conditions existing during the test period;
2) The average total gas volumes varied from 416,000 to 552,000 cfm on furnace no. 1, from 310,000 to 379,000 cfm on furnace no. 2, and from 324,000 to 515,000 cfm for furnace no. 3. (Design volume for each precipitator is 202,000 std cfm with a total of 606,000 std cfm for all three precipitators); after tests, the dampers were adjusted to maintain approximately designed flow through precipitators, regardless of furnace in use;
3) Minimum excess air ratios, occurring at the peak of the carbon evolution, varied from a low of 1.9:1 to 9.3:1 on the heats tested;
4) The average temperature of the gas in the duct just before entering the precipitator inlet header varied from 313° to 507°F for the heats tested;
5) The average humidity of the gas entering the precipitator varied from 3.1 to 20.3 pct by volume; the lower percent moistures occurred on the earlier heats before moisture from the water sprays was supplements with steam; precipitator efficiency tests were run after the percent moisture was increased above the minimum required for adequate fume removal;
6) The rate of carbon burn-off did not form a uniform pattern; for most heats the rate of carbon burn-off increased to a peak approximately ½ to ¾ of the way through the heat and then dropped off rather sharply; in a smaller number of heats the rate of carbon burn-off reached a maximum approximately 1/3 of the way through the heat and remained relatively constant until near the end of the heat and then dropped off; a few heats had carbon burn-off patterns in between these extremes;
7) The dust concentration ahead of the precipitator varied from 2.02 to 4.96 grains per std cu ft and averaged 3.59 grains per std cu ft;
8) The pressure drop through the system was approximately 4.5 in. of water;
9) The pct CO_2 in the gas leaving the furnace mouth varied from approximately 25 pct at the start of the blow to approximately 12 pct near the end of the blow;
10) Precipitator efficiency tests showed dust removal to be greater than 99.8 pct; and
11) The average heat transfer to the hood during the oxygen blow was 535,000 Btu per min with a maximum of 1,029,000 Btu per min as calculated from hood cooling water inlet and outlet temperatures; about 6000 gpm of cooling water is used on each hood.

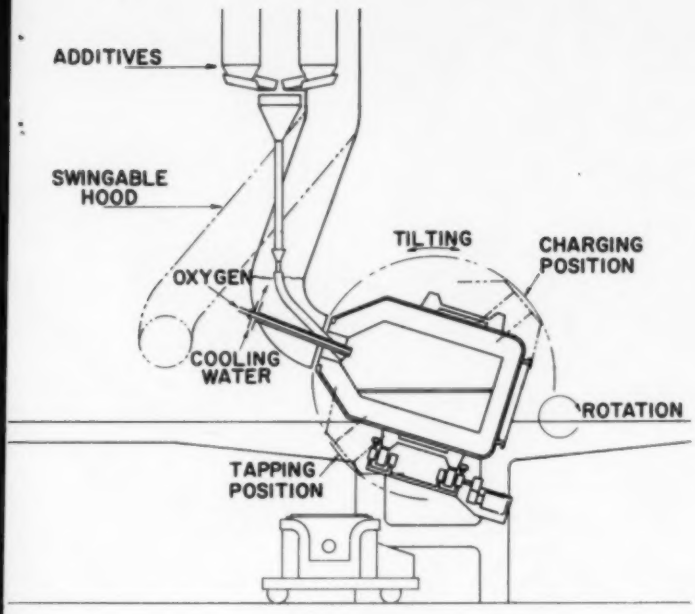
Gas pre-conditioning

Steam is introduced into the furnace gas stream at about 12,000 to 20,000 lb per hr from the start of the blow. There are seven banks of water sprays around the hood to cool the gases and condition them for the precipitator. The first sprays are automatically turned on at 500°F, and the last sprays, at 560°F. The steam is shut off when the third bank of water sprays is turned on and cuts back in again as the gas temperature drops and the third bank of water sprays is shut off. For most of the blow, the gas temperature is between 500° and 550°F at the precipitator inlet. Gas temperatures of over 1000°F have been encountered for very short periods, but the large heat capacity of the precipitator has prevented any damage thus far.

Conclusions

We believe that electrostatic precipitators are a worthwhile investment. And if we were to add another LD basic oxygen shop at Fontana, we would again recommend electrostatic precipitators for the dust cleaning system. However, certain design changes would be made. Some of the discharge electrodes would be replaced and their spacing also changed. Discharge electrodes in the first two sections of each precipitator should have a 10-in. spacing and would be of barbed design. The remaining four sections would be conventional, using plain electrodes and 9-in. spacing.

When one is confronted with a gas cleaning problem like that found in an LD basic oxygen steel plant operated under the previously stipulated conditions, we do not believe that any other available system would present fewer problems or demand less maintenance than electrostatic precipitators.



SHARON STEEL'S KALDO FURNACES

First in US

Sharon Steel Corp. will become the first company in the US to install Kaldo basic oxygen steelmaking furnaces. This article describes the two-furnace facility which is scheduled to be in operation by the Fall of 1962.

by R. N. Merk and D. R. Berg

The long-awaited announcement of the first Kaldo installation for the United States was made earlier this year by James A. Roemer, chairman of the board of Sharon Steel Corp. The two-furnace installation will be designed and constructed by Dravo Corp. and will operate at an initial capacity of about 1-million tons of steel annually. With the addition of a third furnace, the approximate capacity will be increased to 2 million tons.

The decision to install the Kaldo furnaces was made as a result of carefully-controlled quality tests over a full range of carbon steels produced in open-hearth, electric, LD, and Kaldo furnaces. It was concluded that steel produced by the Kaldo method would best supplement Sharon Steel's present range of specialty steels.

The Kaldo basic oxygen steelmaking process was developed by Professor Bo Kalling, director of research for Stora Kopparbergs Bergslags A/B in Sweden. He has been retained by Sharon to serve as a consultant during the construction and initial operation of the new facility.

Kaldo furnaces

In the Sharon installation, the Kaldo furnaces will each have a diameter of 18 ft 1 in. and a length of 26 ft inside the shell; they will be rotated on their longitudinal axes in cradles at variable speeds up to 27 rpm at an inclined angle of 17° to the horizontal. One furnace will operate while the other is being relined. High-purity oxygen will be produced at an on-site 370-tpd plant to be designed and built by Dravo Corp. and owned and operated by the National Gas div. of Chemetron Corp. The oxygen will be injected into the furnace at a pressure of 20 to 45 psig through a 20-ft long, water-cooled, variable position, oscillating lance mounted in the water-cooled exhaust hood.

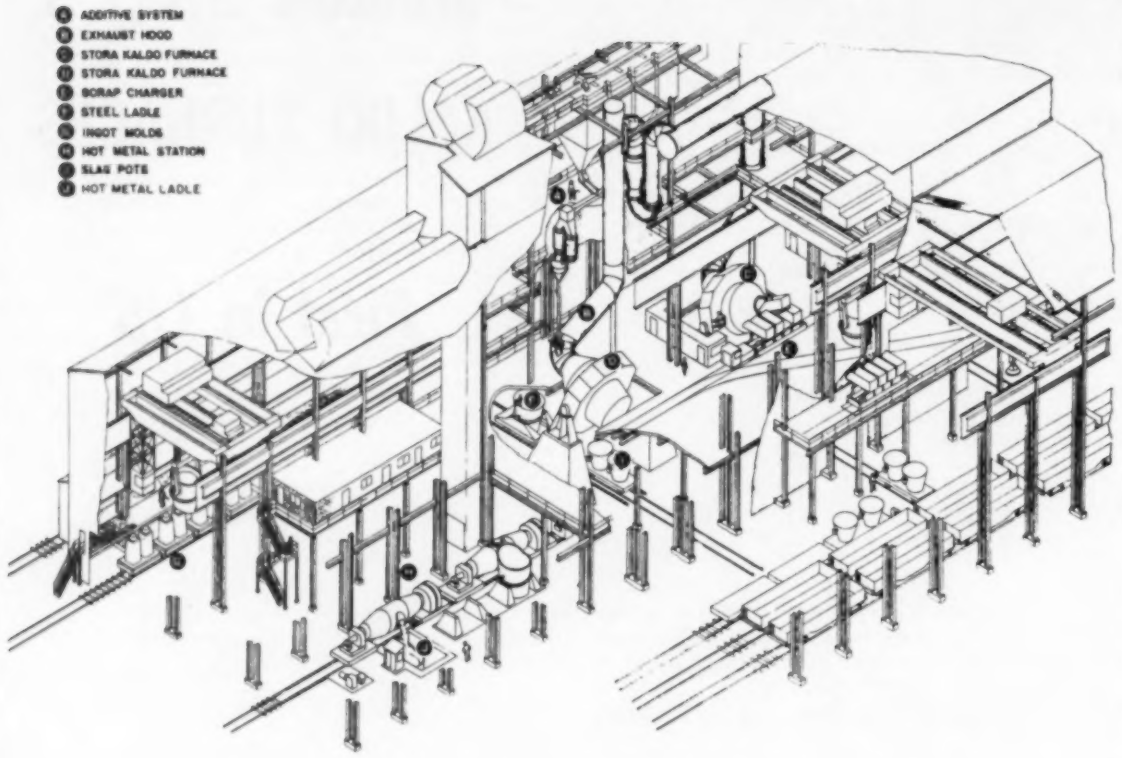
By introducing the oxygen at a low angle to the bath surface without impinging on the molten metal, a slag-metal interface reaction is achieved. This open-hearth type reaction, combined with the rotation of the furnace, provides selectivity in the elimination of metalloids and excellent control of carbon, phosphorus, nitrogen, sulfur, and oxygen, as well as final tapping temperature. The process has an exceptionally high thermal efficiency because virtually all of the carbon monoxide resulting from carbon oxidation is burned to carbon dioxide within the furnace. The heat thus released permits scrap melting of up to 45 pct of the total metallics charged, thus producing 1 2/3 tons of steel per ton of hot metal charged. When ore instead of scrap is used as a coolant, approximately 1 ton of steel per ton of hot metal is produced.

The Sharon vessels will be lined with 220 tons of basic refractory materials capable of withstanding temperatures in excess of 3000°F. The working lining will consist of approximately 160 tons of tar-bonded dolomite-magnesite brick 21 in. thick, plus approximately 4 in. of rammed material of the same composition. Twenty-seven inches of this rammed material will be applied to the removable bottom. A permanent lining of 6-in. thick burned magnesite brick will be installed against the shell.

Plant layout

Sharon's Kaldo steelmaking facility, occupying an area 500 x 244 ft, will be located approximately 1600 ft south of the existing 44-in. blooming mill.

R. N. MERK is vice president, engineering, Sharon Steel Corp., Sharon, Pa., and D. R. BERG is with the Dravo Corp, Pittsburgh, Pa.



Above—Isometric projection of Kaldo installation. Below—Cross-section of the Kaldo furnace, Roemer works, Sharon Steel Corp., Sharon, Pa.

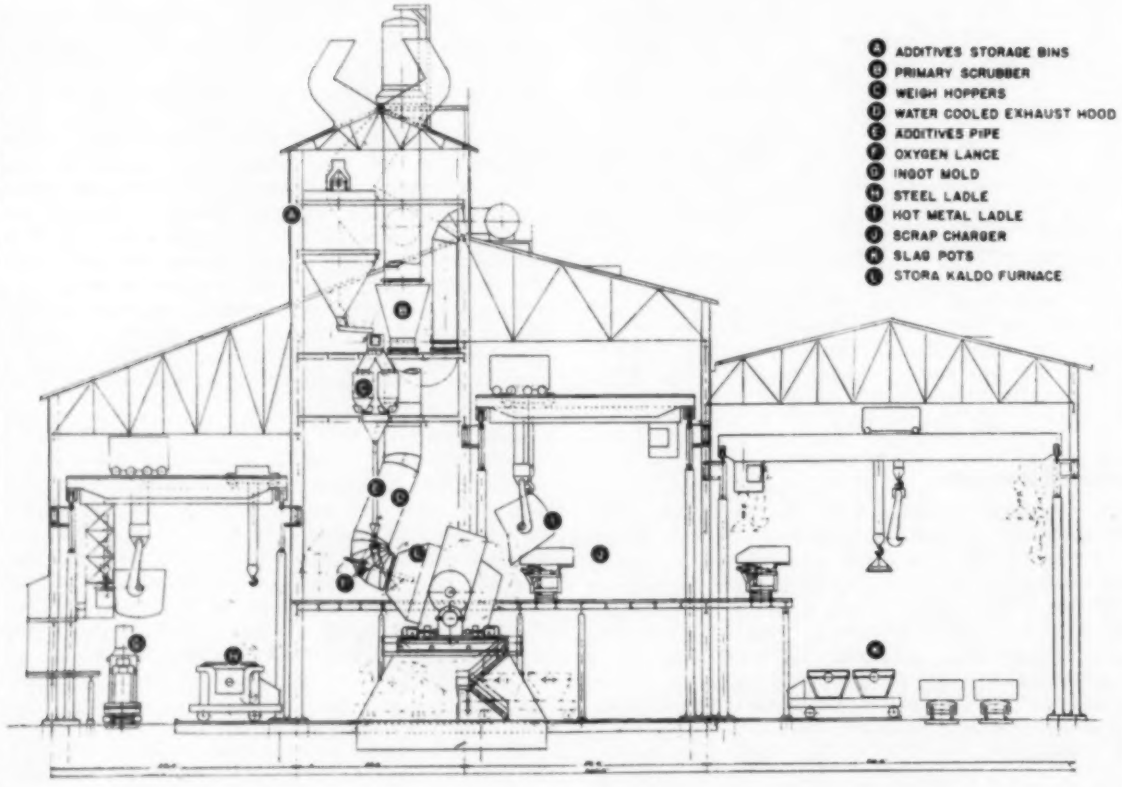


Table I. Kaldo Basic Oxygen Steelmaking Facilities

Company Name	Plant Location	Number of Furnaces	Heat Size, net tons	Operating Date	Normal Metallic Charge
Stora Kopparbergs Bergslags A/B	Domnarvet, Sweden	1*	35	May 1956	High Phos Iron Ore Coolant
SOLLAC	Seremange, Moselle, France	1*	130	April 1960	High Phos Iron Scrap Coolant
Oxelosund Steel Works	Oxelosund, Sweden	2	130	June 1961	Low Phos Iron Ore Coolant
Consett Iron Co. Ltd.	Consett, England	2	130	July 1962	High Phos Iron Scrap & Ore Coolant
Sharon Steel Corp.	Sharon, Pa., USA	2	150	Fall 1962	Low Phos Iron Scrap Coolant
Park Gate Iron & Steel Co. Ltd.	Rotherham, England	2	85	1963	High Phos Iron Scrap & Ore Coolant
Shelton Iron & Steel Ltd.	Stoke on Trent, England	2*	50	1963	High Phos Iron Scrap & Ore Coolant
Stora Kopparbergs Bergslags A/B	Domnarvet, Sweden	2*	70	1964	High Phos Iron Ore Coolant

* With exchangeable vessels.

It will be housed in a structural steel frame building with protective metal cladding and supported on reinforced concrete foundations resting on piling. It will include furnace, charging, and teeming aisles, as well as a covered scrap and slag handling bay.

The furnace aisle will be 450x40 ft and will consist of an operating floor and three flux handling and storage floors located above the ground floor. The two Kaldo furnaces, complete with drives and enclosed operating pulpits, will be located in this aisle. Each pulpit will contain a control center with complete instrumentation. Process control of an advanced design will be incorporated.

Slag pot and teeming ladle transfer car tracks will be at ground level beneath each furnace. Finished heats will be transported to the teeming aisle in steel ladles by wide-gauge self-propelled transfer cars; slag pots will be conveyed from the furnaces to the scrap and slag handling bay in the same manner. Other facilities in the furnace aisle will include furnace hoods, emergency draft stacks, furnace refractory storage, and the stopper rod oven.

The charging aisle will be 500x50 ft. The hot metal will enter the north end of the aisle in 250-ton capacity torpedo ladles and will be poured into 150-ton hot metal ladles at the reladling station. A 200/30 ton four-girder crane will transport the hot metal from the reladling station to the furnace. Ladle skulling and relining facilities will be in the south end of this aisle.

The teeming aisle will be 450x50 ft and will have two teeming platforms, one 225 ft long and the other 150 ft long. The steel ladles will be lifted from the transfer cars by either of two 200/40-ton capacity teeming cranes.

The covered scrap and slag handling yard will be 350x80 ft and will be located adjacent to the charging aisle. Scrap will be brought into this yard in gondola cars and will be transferred to the scrap charging cars or to ground storage by means of a 35/15-ton crane and lifting magnet. This crane will also be used to move the slag pots from the slag pot transfer cars to railroad cars for disposal at the slag dump. There will be track space for approximately 14 cars and a scrap storage bin with a capacity of approximately 1700 tons.

The granular additives—ore and burnt lime—will be unloaded from bottom-dump railroad cars into a covered track hopper at ground elevation. From this point, the additives will be elevated by belt conveyor and distributed by tripper to overhead storage bins having a total volume of 1400 cu ft. This stocking operation will be controlled from a console at the track hopper shelter. The burnt lime and ore will be fed by vibrating feeders and conveyors from the storage bins to two weigh-hoppers above each furnace. From there they will be discharged in weighed quantities into the furnaces through an additive pipe and water-cooled chute through the furnace hood. These additions will be made during the oxygen blow. The entire weighing and feeding operation will be remotely controlled from a console located in the operating pulpits.

Storage-type vibrating feeders for ferroalloy ladle additions will be installed beneath the operating floor at each furnace.

The exhaust gases from the operating furnace will be drawn through water-cooled hoods and ducts to a venturi gas cleaning system by a motor-driven 50,000 cfm induced-draft fan. The clean gases will be discharged to atmosphere through an unlined steel stack. The dust laden water arising from this gas cleaning operation will be pumped to a thickener-vacuum filter installation for further conditioning prior to disposal. An emergency stack will be provided for exhausting fumes to atmosphere in the event of failure of the induced draft fan.

The lower portion of the water cooled exhaust hood, containing the flux additive chute and the oxygen lance, is arranged to swing clear of the furnace for tapping, slagging, charging, and sampling operations.

Other Kaldo installations

While Sharon's will be the first Kaldo steelmaking unit to be installed in the Western Hemisphere, there are three Kaldo facilities now in operation: the original plant at Domnarvet, Sweden; the Sollac plant at Seremange in the Lorraine area of France (see JOURNAL OF METALS, Nov. 1960, pp. 853-856); and the Oxelosund Steel Works at Oxelosund, Sweden (described elsewhere in this issue). Including Sharon, there are five installations in the planning and construction stages. These are shown in Table I.

On SWEDEN'S SECOND KALDO PLANT

On June 13, the first heat was made in a 110-ton Kaldo furnace at Sweden's new integrated steel plant at Oxelosund; the second furnace will go into operation shortly. Operations mark the first commercial utilization of Kaldo for low-phosphorus hot metal.

by Carl Sebardt

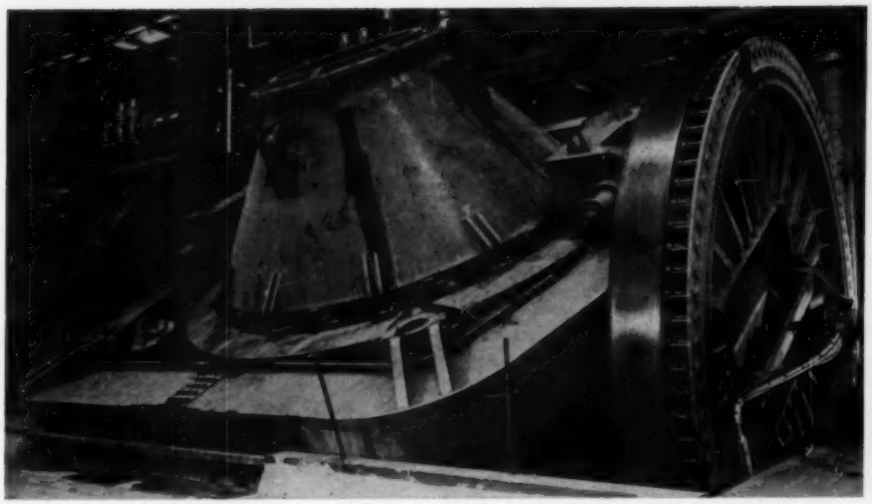
The city of Oxelosund is situated approximately 75 miles southwest of Stockholm, on the Baltic Sea. In the early 1870's, it was still a quiet fishing village when the Grangesberg company, decided to build an export harbor for iron ore which would be the end point of a new railroad from the ore fields in middle Sweden. Through the years, this harbor has been expanded and mechanized, and today over 2 million tons of ore are shipped annually from the mines of Grangesberg and other companies.

In 1912, Swedish and French interests decided to build a small blast furnace and coke plant at Oxelosund. Surplus gas from the blast furnace and coke ovens was later utilized in a window glass plant, which today supplies close to 40 pct of the Swedish consumption. In 1954, a new, larger blast furnace and new coke ovens were put into operation, and the old blast furnace was closed down. Shortly thereafter, the Grangesberg company took over the controlling interest in the Oxelosund iron works.

At that time, the Swedish Government was negotiating for acquisition of the Grangesberg shares in the Lapland iron ore mines. Such a take over by the Government would result in a considerable addition of capital to Grangesberg, and it was felt that a part of this money could eventually be used for expanding the Oxelosund Iron Works to a fully integrated steel plant.

In this manner the ore from the Grangesberg mines in middle Sweden could be refined, and some of the lost sales and profit potentials of the company regained. Market investigations had shown a sufficient need for heavy plate in Sweden to justify the building of a large steel plant for the manufacture of plate.

CARL SEBARDT is managing director of the Oxelösunds Järnverk of the Grängesberg concern at Oxelösund, Sweden.



Opposite page, new Kaldo furnace in operation at Oxelosund, Sweden; at top charging, and at bottom tapping. At right, the furnace prior to start-up.

At the end of 1956, a planning committee from the Grangesberg company in cooperation with the English consulting firm, the International Construction Co., proposed the building of an integrated steel plant with a capacity of 330,000 tons of heavy plate per year. After several revisions, the plans included expansion of the existing harbor and coke plant, construction of a new sintering plant with a capacity of about 2700 tons sinter per day, a new 23-ft blast furnace, and a new melt shop with one 165-ton open-hearth furnace and two 110-ton Kaldo oxygen furnaces. This melt shop was to have an ingot capacity of 475,000 tons per year. The rolling mills would include a slabbing mill and a plate mill of 12-ft width. In addition, an oxygen plant had to be built, as well as maintenance shops, offices, laboratories, etc.

In January 1957, the Board of Directors of the Grangesberg company approved the project which had been calculated to cost about $110 million, including operating capital, start-up expenses, and contributions to the planning and construction of housing. In view of the large investment, it was considered desirable that the construction of the plant be expedited as much as possible so that the plant could be started up in the beginning of 1961 and be in full operation at the end of the same year.

The most interesting part of the committee proposal was to base the main steel production on the Kaldo process. In the Spring of 1956, a 30-ton Kaldo furnace had been started at Domnarvet, and the results had been so good that the committee ventured to recommend this method. The fundamental idea in building the Oxelosund steel plant was to use ore as raw material. Because the Grangesberg company disposed of both high-phosphorus and low-phosphorus ores, a steelmaking method was needed which could utilize both types. The basic-bessemer method was out of question for the manufacture of ship plates, and the LD method, at that time, had not shown any favorable results when using high-phosphorus pig iron. If the Kaldo process would fulfill its initial promises, it would be ideal for the new steel plant. However, an open-hearth furnace was considered necessary for remelting of internal scrap from the rolling mills. At that time, sufficient experience regarding the scrap melting capability of Kaldo had not been obtained.

The melt shop consists of two main aisles 700 ft x 83 ft with a maximum height of 100 ft.

The hot metal is transported from the blast furnace in 100-ton ladles to the mixer which has a capacity of 1000 tons. The mixer is lined with magnesite brick and is heated with coke-oven gas; it is placed in line with the columns between the furnace aisle and the pouring aisle in the middle of the melt shop. Room is provided for one additional mixer.

The hot metal is transported from the mixer to the open-hearth furnace and the Kaldo furnaces in 135-ton ladles after being weighed on a 225-ton capacity hot-metal scale.

The 165-ton basic open-hearth furnace is of Maerz design with an all-basic roof; its calculated production is 145,000 tons of ingots per year. The burners are designed for oil or a combination of oil and coke-oven gas with oxygen addition.

The open-hearth furnace is charged with about 30-pct hot metal from the mixer and the balance scrap from the slabbing and plate mills. Total heat time is about 8 hr. The slag is tapped on the floor and, after solidification, is removed with an excavating machine.

Kaldo plant

The main units of the steel plant are two 110-ton Kaldo furnaces with the following dimensions:

Furnace body, length	26 ft 3 in.
Furnace body, inside diam (barrel)	18 ft
Furnace, diam inside new lining	13 ft 2 in.
Furnace, mouth, diam, new lining	5 ft
Furnace, bottom, diam, new lining	9 ft 2 in.
Rotation rings, outside diam	22 ft 8 in.
Rotation rollers, outside diam	38 in.
Tilting rings, outside diam	22 ft 2 in.
Tilting rollers, outside diam	44 in.
Blowing position	17°–20°
Maximum rotation speed	25 rpm

The rotating furnace body with lining weighs almost 450 tons, including the lining of 280 tons. The refractory lining in the barrel part of the furnace consists of 6 in. of magnesite brick against the shell, plus a working lining of 6 in. rammed tar dolomite and 18 in. of sintered dolomite brick. When the new lime and dolomite plant is ready for operation, Oxelosund will produce its own tar dolomite bricks.

The rotating drive consists of eight 100-kw direct-current motors connected in series with two generators. The maximum tilting speed is 1 rpm; tilting machinery is driven by two 140-kw direct current motors.

The oxygen lance and a lance for continuous additions are attached to the swinging exhaust hood. The maximum oxygen input rate at the start-up was 5600 cfm. The oxygen lance has an inside diameter at the mouth of 5 in. and is provided with a motor drive to give it a sweeping motion.

The 145-tpd Linde-Frankl oxygen plant delivers oxygen of 98 pct purity to the Kaldo furnaces and oxygen of 99.6 pct purity for scarfing, etc.

Lime, iron ore, and other materials are moved by conveyor belt to overhead storage bins; there are four bins for lime, three for ore, and one for return slag. From the storage bins these materials are fed to two weighing hoppers above each Kaldo furnace. The weighing is automatic and is controlled from the operating pulpits. Normally iron-ore concentrate will be used as a coolant, and this is added continuously through the injection pipe during the blow. It can also be added through a chute with the furnace in a non-blowing position; this chute is used for the addition of lime.

The exhaust gases are collected in a water-cooled hood and conducted through a water-cooled pipe to a primary cooler where the gas temperature is reduced to 575°F. The gases are then cleaned in a dry electrostatic precipitator. The gas cleaning efficiency is 99 pct, and the maximum dust loading 0.04 grains per cu ft. The dust is removed from the precipitator by screw conveyors into closed containers and transported to the sinter plant.

The steel is tapped in a 135-ton ladle on a transfer car. In order to avoid excessive splashing during the tapping of the steel, the transfer car is provided with an hydraulic lifting device for the ladle. After transfer to the pouring aisle, the ladle is picked up by one of the 275-ton teeming cranes.

The planned production of the Kaldo plant is 350,000 ingot tons per year.

Maintenance and rebricking of both hot-metal and steel ladles is carried out in the teeming aisle. There are three 200-ton steel ladles for the open-hearth and five 135-ton ladles for the Kaldo furnaces.

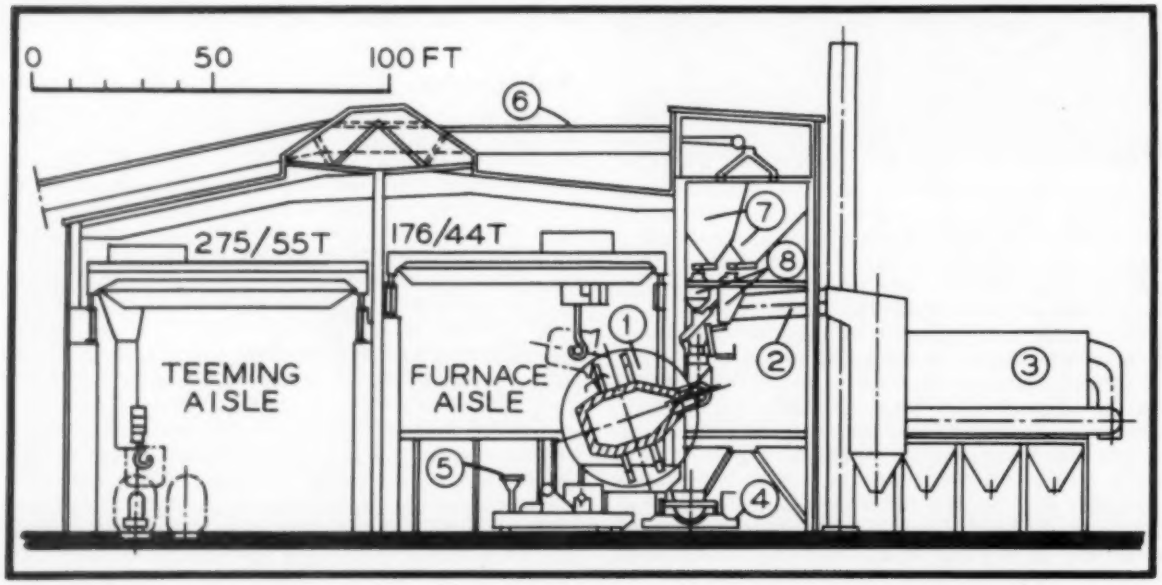
There are two teeming platforms: one for the open-hearth and one for Kaldo. Only top pouring is used, into 11, 19, and 22 ton molds on cars.

Operations

Since the first heat was tapped on June 13 and the steel works has been closed three weeks during July on account of workers' vacations, it is too early to report operating results at this time. Furthermore, the operation during the first two weeks was used to adjust the oxygen plant, the mechanical and electrical equipment of the furnace, instrumentation, and regulating devices which, quite naturally, made normal furnace operation impossible. However, it can be stated at this time that, both mechanically and metallurgically, the installation has functioned according to calculations and in a very satisfactory manner.

The furnace has been charged with an average of 90 tons of hot metal of the following composition: 4.3 pct C, 1.0 pct Si, 1.0 pct Mn, and 0.050 max P and max S. About 20 tons of cold pig-iron scrap has also been charged. It is our intention to use a low-phosphorus hot metal of the above analysis for some time, and later to change to a high-phosphorus hot metal. The coolant has been iron-ore concentrate which has been blown in continuously with compressed air through the injection pipe during the blow.

Output of the steel plant is intended for ship plate. Both killed and semi-killed types have been produced in the Kaldo furnace in the range of 0.12 to 0.15 pct C with about 1 pct Mn. Phosphorus and sulfur have been 0.012 pct and lower. The properties of the finished plates have been quite satisfactory.



Oxelosund steelmaking plant, section of Kaldo area. 1–Kaldo furnace. 2–waste gas pipe. 3–electrostatic precipitator. 4–slag pot transfer car. 5–ladle transfer car. 6–conveyor for lime and ore. 7–storage bins. 8–weighing bins.

The Third ELECTRON BEAM SYMPOSIUM

by Robert Bakish

A summary of the third Alloyd Electronics Electron Beam Symposium held in Boston on March 23 and 24. A full account of the Symposium is to be found in the recently published Proceedings.

The Third Alloyd Electron Beam Symposium was held in Boston as usual, in late March. The attendees included representatives from three continents, five countries, 170 companies, and, with a total attendance of 350, indicated the tremendous increase in interest in this field of technology.

This year's conference consisted of four sessions; these were *The Physics of Electron Beams, Electron Beam Welding, Unusual Applications of Electron Beams,* and *Applications of Electron Beams to Microminiaturization.* Twenty speakers from France, Germany, and the United States participated. This review is an attempt to cover the highlights of each presentation and to convey the readers an idea of the scope and growth of activities in this technology.

The meeting opened with an introductory talk by R. Bakish, symposium chairman, who welcomed the participants and presented the aims of Alloyd's Symposia. In essence, these meetings aim at fostering progress in electron beam technology and encouraging the establishment of informal and personal relations among its pioneers.

Physics of electron beams

Sessions started with a talk by Professor L. D. Smullin of MIT entitled *Introduction to the Physics of Electron Beams.* The underlying principles of electron guns and the beams they generate were discussed, using a rather fundamental approach. Professor Smullin directed his talk toward both the neophyte and the experienced electron optics physicist.

John R. Morley of High Voltage Engineering Corp. followed with a timely talk on electron gun design and cathode materials limitations. Extensive experimental data on emission characteristics, evaporation rates, life of cathode materials on one hand, and fundamental factors concerning power density and beam diameters on the other, were presented.

M. H. Hablanian of NRC Equipment Corp. presented some information on the still-controversial subject of the reasons for large penetration ratios in low-voltage systems. His paper was based on data obtained with an indirectly heated filament Pierce-type gun and its performance as a welding device.

L. A. Harris of the General Electric Research Laboratory presented a summary of his recent work with hollow electron beams. His paper brought out very interesting and worthwhile information on this lesser-known type of electron beam configuration and the different methods of uniformly focusing dense, hollow cylindrical beams. Both torroidal and variable perveance guns were discussed in detail. Fig. 1 shows schematically one of the systems for the generation of hollow beams. With some imagination, one could see in the not-too-distant future, the utilization of hollow beams as the means of welding circularly symmetrical components.

Dr. Harris' discussion was followed by a joint presentation by J. W. Lotus and R. C. Gunther from the Raytheon Research Div.; they discussed the utilization of computers for electron beam trajectory calculations. This mathematical presentation showed some of the complexities of the electron optics problems that workers in the field face and the great assistance that computers may lend to this technology.

The last paper in this session, prepared by R. Connor of High Voltage Engineering Corp. deviated from the main topic of the meeting since it discussed the physics of ion beams. With the growth and acquisition of knowledge in the field of electron beams, it appears that ion beams also will be playing an important role in the future.

The presentation reviewed the many different ion sources that have been used and discussed the presently-existing designs. It emphasized the duoplasmatron ion source of von Ardenne. The increased influence of positive ions leads to considerably greater difficulties in focusing and brings forth a degeneration of the ion optical system. This presentation completed the session on the *Physics of Electron Beams.*

Electron beam welding

The second session, which was devoted to electron beam welding developments, asserted the rapid growth in 1960 and indicated the establishment of

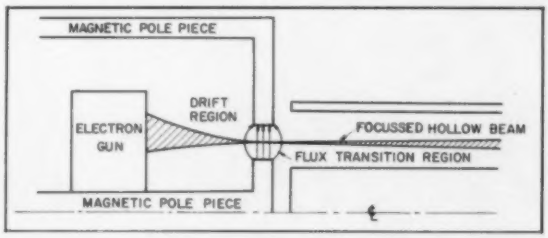


Fig. 1—Idealized hollow-beam injection system showing the separate regions.

ROBERT BAKISH is director of research, The Alloyd Corp. and vice president of The Alloyd Electronics Corp., Cambridge, Mass.

electron beam welding as a joining technique to be heard of with increased frequency along with the more conventional joining methods of today.

The audience was fortunate to have as the first item on the program a talk prepared by J. A. Stohr of the French Atomic Energy Commission, who having started work in 1957, is one of the pioneers in electron beam welding. Dr. Stohr in his paper discussed the scope of electron beam welding in France and its application to welding metals and alloys in the nuclear power field. New guns and welding systems configurations developed for the Saclay Atomic Energy group were discussed. Fig. 2 shows a special design of a moving electron gun used by this group in France. The French electron beam welding effort is exclusively in the low-voltage field.

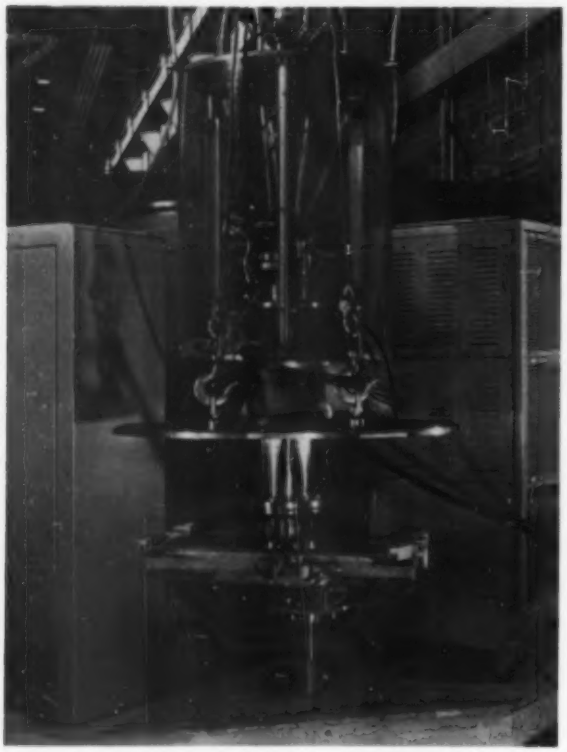


Fig. 2—Special design for moving electron gun.

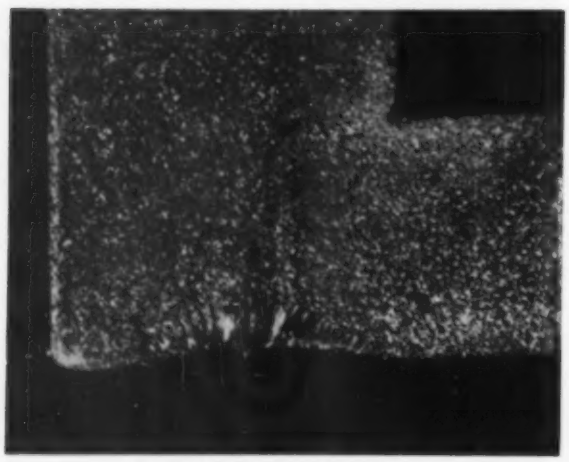


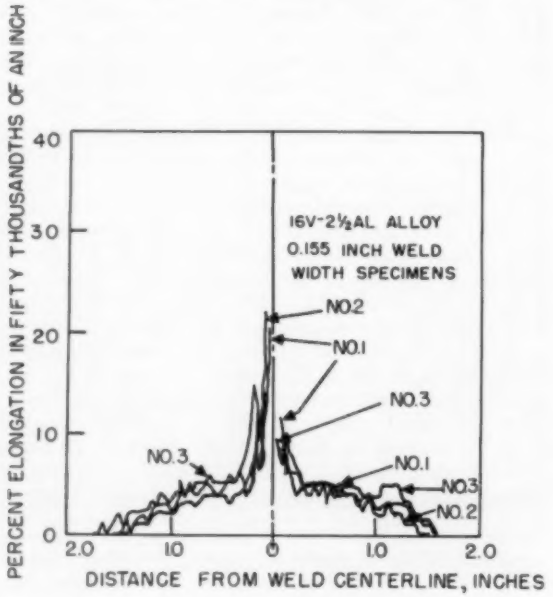
Fig. 3—Above, Superficial weldment of beryllium. Fig. 4—Right, Incremental strain percentages for three specimens representing one welding condition.

The next paper was presented by H. H. Hoffman of the Olin Mathieson Chemical Corp. Data on electron beam welding with a high-voltage system were presented. Materials processed in the course of these studies were zircaloy; Inconel; stainless steels; aluminum; 1100 and 2024 aluminum alloys; columbium; and tungsten.

John Meier from Hamilton Standard presented an extensive compilation of electron beam welding data from high-voltage machines and an evaluation of physical properties of electron beam weldment in 302 stainless, 4340, 17-7PH stainless, 6Al-4V titanium, and 61S Al. Data included tensile and fatigue properties. In addition to mechanical property data, Mr. Meier presented some interesting data on the depth of penetration as function of thermal conductivity for these alloys.

These discussions were followed by two presentations by W. T. Hess and H. J. Lander of the Alloyd staff who, along with Mr. Hablanian of NRC, the Saclay group, and others, employ the low-voltage approach. The first presentation, on electron beam welding of beryllium given by Mr. Hess, discussed the great strides made by electron beam welding for joining *space-age* metal. Two particularly important facts appeared in Mr. Hess' presentation. First, for the most part, electron beam beryllium weldments developed by this group are of almost equivalent strength to that of the parent metal—a finding which could open new vistas for joining of this metal. The second fact is the ability to produce surface welds with shallow penetration thus permitting the utilization of this technique for encapsulation. Fig. 3 shows one of the superficial weldments.

The presentation on electron beam welding of rocket casing materials by Mr. Lander presented some very sound and systematic data on heat-affected zones, strain distribution, and tensile strength of electron beam weldments of titanium alloys. Of particular importance is the great attention devoted to the measurements of the true strains and the distribution of these in the vicinity of the



weldment areas rather than just considering average strains associated with the weldments. Retention of useful properties in all these materials speaks a great deal in favor of low-voltage welding. Fig. 4 shows a typical strain distribution curve for a titanium alloy.

Unusual applications

The next session on the *Unusual Applications of Electron Beams* presented information on seldom-heard-of uses of electron beams. It started with two papers discussing beams of considerably higher power than those discussed in previous presentations. These beams are of the linear Van de Graaff accelerator type with voltages up to 1 million v.

E. F. Degering presented results of studies made with his associates on the utilization of these beams as radiation sources and their use as polymerization promoters and means for altering materials. A great variety of materials have been studied and results reported. Data on the role of radiation in polymerization of some organic chemicals is presented in Fig. 5.

Dr. Degering's presentation was followed by a talk by Charles Merritt, also a member of the Quartermaster Research and Engineering Laboratories. In this presentation, the means available for study of electron beam radiation—induced changes on these materials—were discussed. It was stated that volatile components generated as a consequence of radiation changes in a sample are collected by vacuum distillation procedure. The condensates of these extracts are fractionated by low temperature vacuum distillation, gas chromotography, or both, and the components are finally identified by mass spectrometry. Results from these studies were presented.

The next presentation by R. E. Ogilvie of MIT presented details on the most interesting technique of the electron probe microanalyzer and the type of data that can be obtained with this very powerful research tool. After presenting the fundamentals of the system and the mechanisms of its operation, Professor Ogilvie proceeded to illustrate the great versatility of the system and its application to specific problems.

K. H. Steigerwald of Zeiss, a distinguished and early worker in this field (initial activities as far back as 1938) who continues to be active today and who has made and continues to make substantial contributions, was our second overseas speaker. He reviewed briefly the historical development of the electron beam devices leading to the present family of devices manufactured by his company. The emphasis of his talk was placed on the fundamentals underlying the approach utilized in the Zeiss' systems and provided data on the resolution capabilities of micro-machining or electron beam milling as he refers to it. Figs. 6 and 7 show results of slot milling in the 10 micron range and the appearance of the milled surface as observed with an electron microscope. Energy densities achieved with electron beams and other systems are also compared by Mr. Steigerwald in Table I which gives values on minimum cross section possibilities and maximum power densities achieved with these techniques.

Table I. Two Heat Sources Compared with Electron Beam

Heat Sources	Smallest Cross Section	Highest Energy Density
Oxyacetylene Burner Flame	10^{-2}	10^{4}
Carbon Arc	10^{-3}	10^{5}
Electron Beam	10^{-7}	10^{9}

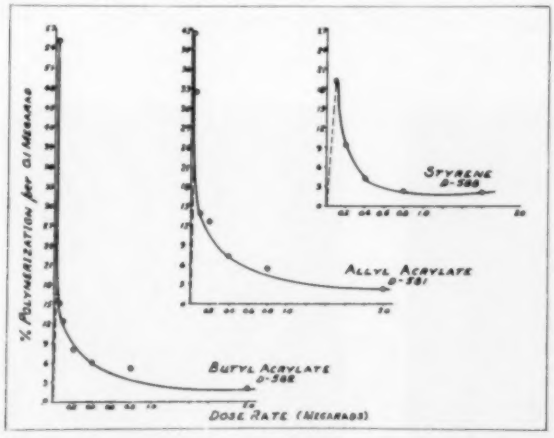


Fig. 5—Effect of dose rate on allyl acrylate, butyl acrylate, and styrene.

Microminiaturization

The last session considered the very timely topic of *Microelectronics* and demonstrated the role which

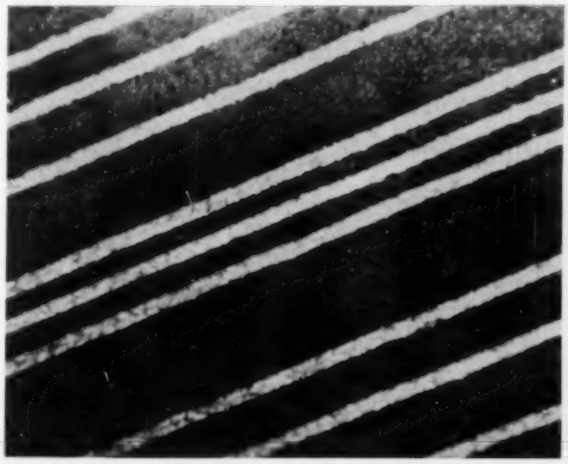


Fig. 6—Above, Highly magnified slots on wafer resistor with a slot width of 10 μ. Fig. 7—Below, Electron microscopic picture of slot milled by electron beam on the surface of aluminar ceramics. 4420X. Reduced approx 50 pct for reproduction.

electron beams will play in it. O. C. Wells of Westinghouse started the session with an excellent and comprehensive review of electron beams, their purpose, their capabilities, and their long range prospects related to microminiaturization. The great vistas that are opening to us as well as the array of difficulties that we face were discussed in great detail. The beams with improved resolution are to serve as one of the basic tools in the development of this highly sophisticated field of microelectronics.

The second paper in this session presented by H. R. Poppa of Convair Astronautics discussed the uses of electron microscopes as a means for thickness measurements of electron transparent films. Calibration procedures, the accuracy of this technique (which is of the order of 1 pct for films down to 100 Å thick), and its applications, were discussed. Ability to resolve mono-layers was demonstrated.

The great future prospects of electron beams for microminiaturization and micro-recording were also dramatically illustrated by our third overseas guest, G. Möllenstedt of the Institute for Applied Physics of the University of Tübingen in West Germany. Professor Möllenstedt, who reported on work in the University, demonstrated abilities to resolve recorded information down to 5Å level. With miniaturization of the order seen in Fig. 8, one could envisage recording the text of the Bible on a fraction of the area of a postage stamp. He apprised the audience that, although these techniques are capable of these resolutions and recording information at high densities, there are still many problems associated both with the recording and the reading of this information at the present state of the art. Electron microscopes are the sole means of reading these high density messages today, but the future of this activity is certainly bright and exciting.

In the quest to produce high-purity thin films for high-reliability electronics components, a movement toward the utilization of electron beams as heat sources for evaporation is clearly seen today. The main virtue of electron beams as heat sources for this purpose is their ability to evaporate almost all of the presently known materials by means of high localized temperatures. R. W. Berry of the Bell Telephone Laboratories presented results of his research in the production of thin film tantalum resistive and capacitive components utilizing electron beam evaporation. In his studies, Dr. Berry used electron beams of relative simplicity compared to the more sophisticated beams discussed in connection with microminiaturization. His apparatus used a simple annular filament-type gun.

The concluding lecture by W. E. Flynt of the Varo Manufacturing Co. of Garland, Texas, was on an ion beam system for micro circuitry production; it reminded the audience that both electron and ion beams would be sharing the burdens of the future in this field of microminiaturization. A schematic drawing of the ion beam gun which Dr. Flynt is developing for his work can be seen in Fig. 9. The great variety of experimental problems that were encountered by Dr. Flynt—some of them already mentioned by Mr. Connor in the first session—were discussed in detail.

Among the most important problems to be resolved are the deflection and focusing of the ion beams and the problem of avoiding excessive impingement of the ions on the substrate. Unless the latter can be avoided, a sputtering reaction takes place and the material which is under bombardment is sputtered in a cloud. One last problem that faces ion beams in this application is their neutralization. Assuming these difficulties to be surmountable, the ion source discussed and ion sources in general have some important long range possibilities.

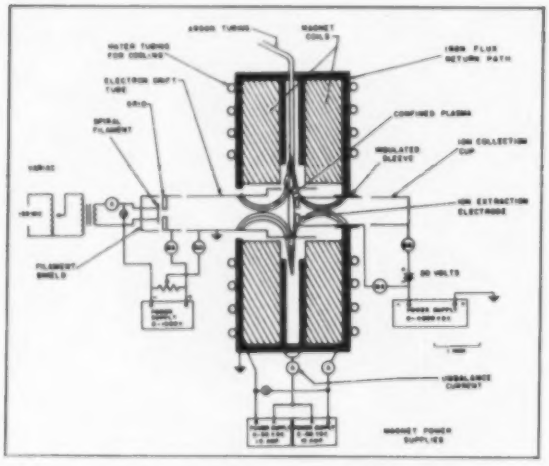


Fig. 9—Schematic diagram of experimental ion source.

The symposium *Proceedings* have been edited and are available for $15 (See JOURNAL OF METALS, Aug. 1961, p. 519) from Alloyd Electronics Corp.

Fig. 8—Capital letters of 0.5 μ size recorded on collodion foil with the electronprobe micro recorder.

AIME STAFF:

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ASST. SECRETARIES—J. B. ALFORD, H. N. APPLETON, J. C. FOX, R. W. SHEARMAN, C. J. HICKS

ASST. TREASURER—JOHN LYNCH

METALLURGICAL SOCIETY STAFF:

SECRETARY—R. W. SHEARMAN

ASST. SECRETARY—D. C. JOHNSTON

NEWS OF YOUR PROFESSION

JOINT SESSION WITH ASM WILL OPEN METALLURGICAL SOCIETY'S FALL MEETING

A *Joint Session on Professional Activities* will be the opening feature of The Metallurgical Society's Fall Meeting which will be held in Detroit October 23-26 at the Pick-Fort Shelby Hotel.

The joint session will be held on Sunday, October 22 at 8 pm in the Michigan Room of the Statler Hilton Hotel. It will be jointly sponsored by The Metallurgical Society's Committee on the Metallurgical Profession and the American Society for Metals' Development Committee. Co-chairmen of the session will be W. R. Hibbard Jr., of General Electric Co., and W. D. Manly, Oak Ridge National Laboratory.

The program will consist of two talks: *The Program of the Metallurgy and Ceramics Foundation* by George A. Roberts, Vanadium Alloy Steel Corp.; and *The Question of Establishing a National Academy of Engineering*, by H. K. Work, New York University.

The Fall Meeting will be held in conjunction with ASM's Metals Show and Exposition which will be held at Cobo Hall.

The Institute of Metals Division will sponsor two symposia of particular interest. The first will be on *High Purity Iron and its Dilute Solid Solutions*, which will be presented on Monday, October 23. One day of technical sessions will be devoted to a discussion of fundamental properties, rather than the engineering aspects, of high purity iron and dilute solid solutions.

The other IMD symposium will concern *Effects of Surface and Environment on Strength*, and will take place all day on Monday and on Tuesday morning. The program will deal with the flow and fracture of metals and ionic solids as affected by environment (gaseous environment, liquid metals, surface-active media, aqueous media), by solid surface coatings, or by surface treatment (e.g. by dissolving the surface during the test).

The Extractive Metallurgy Division will present four technical sessions during the meeting. Two, on Tuesday afternoon and Wednesday morning (October 24-25), will deal with *Vacuum Techniques in Metallurgy*. The other two sessions, devoted to *Nuclear Fuel Reprocessing*, will be presented on Wednesday. The first session will concern *Pyroprocessing*, and the second will cover *New Foreign and American Developments*.

The Iron and Steel Division will present two technical sessions during the Fall Meeting. A session on *Continuous Casting* will be presented in the Ballroom of the Pick-Fort Shelby on Tuesday morning. An afternoon session on *Working of Cast Structure* is also scheduled.

During the Fall Meeting, the AIME Board of Directors will hold an all-day meeting Wednesday, October 25 in the River Room. This meeting is open to any members who wish to attend.

The River Room will also be the scene of The Metallurgical Society's Board of Directors Meeting on Tuesday morning. This will be a Breakfast Meeting.

The Registration area for the Fall Meeting will be located on the Mezzanine Floor between the Ballroom and the Shelby Room.

Thin Film Symposium

October 30 is the deadline for the receipt of abstracts of papers to be considered for presentation at a symposium on the *Properties of Thin Films* which will be presented during the 1962 AIME Annual Meeting in New York. The symposium will be sponsored by the IMD Chemistry and Physics of Metals Committee.

In addition to a session of invited talks, time will be available for presentation of a limited number of papers on thin film research. Prospective contributors are invited to forward abstracts (three copies) and information sheets giving the name and address of all authors no later than October 30, to the Symposium Chairman:

Prof. Charles Elbaum
Richardson Hall
Brown University
Providence 12, Rhode Island

Chauncey D. Leake

Chauncey D. Leake Will Be Main Speaker At Fall Meeting Dinner

Chauncey D. Leake, professor of pharmacology at Ohio State University, will be the principal speaker at The Metallurgical Society dinner during the Fall Meeting in Detroit October 23-26. The dinner will be held in the Terrace Room at the Pick-Ft. Shelby Hotel on Tuesday evening, October 24.

Professor Leake will speak on *Standards of Measurement and Nursery Rhymes*. His talk will deal with the background of standards of measurement, which are the basis for our enormous scientific and technological advance.

In addition to his teaching activities, Professor Leake is chairman of the board of directors of the American Association for the Advancement of Science.

Born in Elizabeth, N. J., Professor Leake received his Ph. D. from the University of Wisconsin in 1923. Before assuming his present position in 1956, he was executive vice president in charge of the medical branch at the University of Texas.

Prior to The Metallurgical Society dinner, the Detroit section, AIME, will sponsor a social hour in the Terrace Room.

Metallurgical Society Initiates Fellow Class of Membership

The Board of Directors of AIME have approved the plan of The Metallurgical Society to establish the *Fellow* class of membership in the society. The new classification will honor members of The Metallurgical Society who have attained distinction as an eminent authority in some aspect of the broad field of metallurgy. A candidate shall have been a full member of AIME for at least five years.

A two-thirds vote of the Board of Directors shall be required for elevation to the class of Fellow. No more than five Fellows will be elected each year, and the maximum number of living Fellows shall not exceed 100.

The Fellows Nominating Committee shall consist of five Fellows, one appointed each year for a period of five years by the incoming President of the Society, and the three most recent living past presidents of The Metallurgical Society. Any member of the Fellows Nominating Committee or any five members of the Society may propose a members name to the Fellows Nominating Committee. There will be no special dues for Fellows.

In order to initiate the Fellow grade of membership, a maximum of 20 will be selected in 1961 by a special nominating committee to be appointed by the Board of Directors. In subsequent years, the normal procedure of not more than five appointments per year will be followed. The Fellows will establish their own procedures for the administration of their affairs.

Proposed Bylaw Change

At the June 20 meeting of the AIME Board of Directors, the following proposals were made for changes in the Bylaws.

Current AIME Bylaws provide that a Member be carried on the rolls for two years after his dues become due. Relatively few Members pay their dues during their second year of delinquency, yet the cost to the Members in Good Standing to carry these Members a second year is appreciable. It is proposed to revise AIME Bylaw **Article II, Section 4.b** from "unpaid for two years" to "delinquent for one year" to provide that Members be dropped one year after their dues become due, effective December 31, 1961.

The AIME Board of Directors voted to change the title of the *Secretary* of AIME to *General Secretary* of AIME. It is proposed to amend the Bylaws to this effect wherever applicable.

Annual Meeting Abstracts

October 30 is the deadline for receipt of abstracts to be considered for presentation at IMD-sponsored sessions during the 1962 AIME Annual Meeting, which will be held in New York, February 18-22.

In order to be considered for inclusion in the program, each submission should consist of the following:

1) Three copies of a 200-300 word abstract, typed double-space on one side only. (No photographs, figures, tables or diagrams, please; however, brief acknowledgments of research sponsorship may be included if applicable.)
2) One copy of an information sheet containing complete mailing address of each author together with title of paper being submitted.

Submissions should be forwarded to: D. C. Johnston, Secretary, IMD, The Metallurgical Society of AIME, 345 East 47th Street, New York 17, N. Y.

Cleveland NOHC Plans Fall Meeting September 21-23

The Cleveland section, National Open Hearth Steel Committee, will hold its fall meeting September 21-23 at the Sheraton-Cleveland Hotel.

About 250 steel men from midwestern and eastern steel plants will attend to hear eight papers on various phases of the oxygen steelmaking process and other aspects of steelmaking.

Friday, September 22, will be devoted to an all-day technical meeting. A highlight of the conference will be the Dinner that evening. D. R. Loughrey of Jones & Laughlin Steel Corp. will be toastmaster. The speaker will be Dr. Carl Beyers, formerly superintendent of schools at Parma, Ohio.

On Saturday morning there will be a plant trip to the basic oxygen furnace shop at Jones & Laughlin's Cleveland plant.

Also on Saturday there will be a meeting of the Board of Directors of the NOHC.

E. J. Sobey of Jones & Laughlin Steel Corp., has been in charge of arrangements for the meeting. Leo Svete of National Tube Co., will assume the duties of chairman at the meeting. He will replace Edward J. Kondas of Republic Steel Corp., as section chairman.

Iron & Steel Institute To Visit U.S.; Attend Society's Fall Meeting

The new Engineering Societies Building will be the scene of the Inaugural meeting October 19 of the Iron and Steel Institute (of London) during its visit to the US. The British group will be in the US October 17-November 7 as guests of The Metallurgical Society and the American Society for Metals. They will attend the Fall Meeting of The Metallurgical Society in Detroit October 23-26.

The meeting on Thursday, October 19, will be presided over by J. S. Smart, Jr., President of The Metallurgical Society, who will make Introductions and Welcoming Remarks. R. R. McNaughton, President of AIME, wil make the Welcoming Address, and Sir Charles Goodeve, president of the Iron and Steel Institute will reply.

The principal speaker will be J. B. Austin, administrative vice president, research and technology, U.S. Steel Corp. Dr. Austin, who is organizational chairman for the visit, will discuss *Three Centuries of Progress in the American Steel Industry.*

The British group will make plant visits and sightseeing tours in the U.S. and Canada during their stay. The itinerary for the entire visit was published in the May issue (page 380) of JOURNAL OF METALS.

Spectroscopy Conf. Planned March 5-9 In Pittsburgh

The 13th Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy will be held at the Penn-Sheraton Hotel in Pittsburgh, Pa., March 5-9, 1962. The meeting will be sponsored by the Analytical Chemistry Group of the Pittsburgh section of the American Chemical Society and the Spectroscopy Society of Pittsburgh.

Symposia are planned on Chemical Analysis of Metals, Gas Chromatography, Raman Spectroscopy, Spectroscopic Studies of Polymers, and Vacuum Spectroscopy. Original papers on all phases of analytical chemistry and spectroscopy are invited.

The number of papers included in the program will be limited, and all papers will be evaluated by the program committee before acceptance. The final deadline for receipt of abstracts is October 16, 1961.

Correspondence regarding papers should be submitted in duplicate to Dr. C. F. Glick, Program Chairman; The Pittsburgh Conference; Applied Research Laboratory; U. S. Steel Corp.; Monroeville, Pa.

DETROIT . . . 1961 FALL MEETING

You'll be visiting:

1. Pick-Fort Shelby Hotel
2. Cobo Hall
3. Detroit River
4. New Detroit Post Office
5. Fort Street
6. Lafayette Boulevard
7. Sheraton-Cadillac Hotel
8. Statler Hilton Hotel

News of the Institute of Metals Division

Advance Registration For Fall Meeting

In addition to an unusually comprehensive technical program, this year's Fall Meeting of The Metallurgical Society, scheduled for October 23-26 in Detroit, boasts increased convenience for registrants in a number of respects. This became apparent when J. Harry Jackson, 1961 chairman of the Institute of Metals Division, announced that arrangements had been concluded making it possible for persons to register in advance to attend sessions scheduled at the Pick-Fort Shelby Hotel.

In prior years, those attending the Society's Fall Meeting, sponsored by IMD and held in conjunction with the ASM Metal Show, had to brave congested registration desks and long lines before they could enter rooms where presentations were often already in progress. With registration totals at the meeting approaching 1500 in recent years, it became obvious that some easing of the registration bottleneck was essential.

The result was a special mailing to all Society members in late August. Included in the preliminary registration packet were a summary of technical sessions planned for the October gathering in Detroit, a preview of the Fall Dinner, and a form on which members could register, as well as reserve tickets for the Dinner, always a highlight of the Fall Meeting. Through a cooperative arrangement with the American Society of Metals, an arrangement was effected whereby potential Metal Show attendees could include the fee for Fall Meeting registration.

Location of Fall Meeting headquarters in the Pick-Fort Shelby Hotel will result in added convenience for registrants who desire to inspect the latest exhibits at the Metal Show. The Exposition will be in Cobo Hall, two and one-half blocks from the hotel.

1962 IMD Lecture By Russian Scientist

Academician G. V. Kurdjumov, head of Metal Physics and Metallography in the USSR's Central Scientific Research Institute for Ferrous Metallurgy, has accepted an invitation to deliver the 1962 Institute of Metals Lecture during the AIME Annual Meeting in New York, Feb. 18-22, 1962. He has not yet decided on the title of his talk, which is scheduled for presentation on Tuesday morning, February 20, in the Astor Hotel.

Dr. Kurdjumov's presentation will be the forty-first in an unbroken series of annual lectures begun in 1922. Arrangements for the 1962 Lecturer's visit to the US are being coordinated by Prof. Morris Cohen of Massachusetts Institute of Technology, a past chairman of the IMD Lecture Committee.

Semiconductors Conf. In Philadelphia, '62

Continuing its effort to bring high quality technical information to members throughout the US, the IMD Semiconductors Committee has scheduled its 1962 Conference on Metallurgy of Semiconductor Materials in Philadelphia. This highly successful series of annual conferences, begun in Boston in 1959 and repeated there in '60, made its west coast debut this year in Los Angeles, where the Metals Branch of the AIME Southern California section hosted the meeting at the Ambassador Hotel from August 30 to September 2.

The Philadelphia section, AIME, is cosponsoring next year's meeting, which is scheduled for the Benjamin Franklin Hotel, August 27-29. Cooperating in arrangements for the meeting in the City of Brotherly Love are: Francis J. Donahoe, The Franklin Institute; A. L. Jamieson, Frankford Arsenal; Edward Korostoff, University of Pennsylvania; F. L. Vogel, Jr., Radio Corporation of America; and Roman J. Wasilewski, E. I. DuPont deNemours & Co.

Bylaws Changes

Revision of the IMD Bylaws concerning the annual lecture sponsored by the Division was accomplished in February, 1961, with results which include a change in name from Annual Lecture to Institute of Metals Lecture, as well as a streamlining of the mechanism for selection of lecturers and arranging for their presentations. The new bylaw appears below:

Article VIII, Section 8—The Lecture Committee shall consist of four members of the Division, one appointed each year for a term of four years by the Chairman of IMD, subject to the approval of the Executive Committee. The member in his second year of service shall be Chairman of the Lecture Committee.

a. It shall be the responsibility of the Lecture Committee to select an outstanding scientific leader to present a lecture on a technical subject of particular interest to the Division at a meeting approximately two years following the time of his selection. The selection is subject to the approval of the Executive Committee, and the Division Secretary shall promply thereafter report the action to the Programs Committee. The lecturer-nominee shall be notified by written invitation prepared by the Division Secretary and signed by the President of The Metallurgical Society at least 18 months prior to the date of the lecture.

b. The Lecture Committee shall be responsible for the arrangements of the lecture presented during its year of office. It also shall be responsible for the itinerary of the lecturer. Financial arrangements shall be subject to the approval of the Budget Committee. An appropriate citation shall be prepared by the Committee for presentation to the lecturer. The lecture shall be known as the Institute of Metals Lecture and the lecturer shall be known as the Institute of Metals Lecturer for that year.

Officers and Committee Members

INSTITUTE OF METALS DIVISION

J. H. Jackson, Chairman
T. A. Read, Past Chairman
David Swan, Senior Vice-Chairman
R. L. Smith, Vice-Chairman
D. C. Johnston, Secretary-Treasurer

Executive Committee: W. A. Backofen, '64, A. T. Cape, '63, M. E. Fine, '62, N. J. Grant '62, J. J. Harwood, '64, J. H. Keeler '62, C.L. McCabe, '63, N. E. Promisel, '63, F. L. Vogel, '64.

Standing Committees

Budget: David Swan, Chairman, J. A. Fellows, J. H. Keeler, O. T. Marzke, R. L. Smith.

Lecture: C. S. Barrett, Chairman, M. E. Fine, R. E. Mehl, E. E. Schumacher, K. R. VanHorn.

Mathewson Gold Medal: J. J. Harwood, Chairman, P. G. Shewmon, C. S. Smith, R. A. Swalin, J. Weertman.

Membership: Alan Lawley, Chairman, W. J. Childs, J. B. Cohen, H. Conrad, J. A. Fellows, J. B. Newkirk, W. G. Pfann, J. W. Pugh, J. Washburn, A. R. C. Westwood.

Nominating: T. A. Read, Chairman, W. J. Harris, Jr., O. T. Marzke, Marvin Metzger, H. W. Paxton.

Programs: R. L. Smith, Chairman, J. J. Harwood, (Fall Meeting), Sheldon Weinig, (Annual Meeting), Chairmen of Technical Committees.

Publications: L. L. Seigle, Chairman, J. N. Hobstetter, Vice Chairman, I. L. Bessen, J. O. Betterton, Jr., R. B. Gordon, R. W. Guard, M. Herman, P. G. Shewmon, C. D. Starr, R. S. Davis, J. Gurland, R. Nash, T. N. Rhodin, L. Slifkin, J. K. Stanley, H. H. Johnson, Jr., J. J. Heger, C. D. Graham, Jr., P. A. Beck, J. B. Clark, A. E. Dwight, R. W. Fountain, E. L. Harmon, A. A. Hendrickson, R. H. Richman, William Rostoker, C. W. Spencer.

Scope: J. H. Keeler, Chairman, J. A. Fellows, Vice Chairman, Chairmen of Technical Committees.

Alloy Phases: P. A. Beck, Chairman, H. Beattie, J. O. Betterton, A. A. Burr, J. B. Derby, Jr., L. S. Darken, Pol Duwez, E. A. Dwight, Reed Elliott, R. P. Elliott, W. L. Fink, R. W. Fountain, K. A. Gschneider, Michael Hoch, H. Y. Hunsicker, R. I. Jaffee, J. S. Kaspar, T. B. Massalski, Harold Margolin, M. V. Nevitt, E. Parthe, R. H. Pry, J. A. Rayne, T. J. Rowland, F. N. Rhines, H. H. Stadelmaier, J. M. Sivertson, A. Taylor, J. E. Westbrook, C. W. Spencer.

Chemistry and Physics of Metals: M. E. Fine, Chairman, P. A. Beck, C. E. Birchenall, S. S. Brenner, L. S. Darken, R. S. Davis, Charles Elbaum, J. J. Gilman, J. N. Hobstetter, Larry Kaufman, J. B. Newkirk, A. S. Nowick, G. M. Pound, R. A. Swalin, J. H. Wernick, C. A. Wert, T. N. Rhodin, Morris Tanenbaum.

Corrosion Resistant Metals: J. J. Heger, Chairman, J. E. Draley, E. J. Dulis, M. G. Fontana, E. A. Gulbransen, A. J. Lena, Marvin Metzger, J. W. Mitchell, R. J. Raudebaugh, S. J. Rosenberg, L. R. Scharfstein, D. E. Thomas, W. E. Tragert.

Electrical and Magnetic Metals: Anthony Arrott, Chairman, R. G. Aspden, B. L. Averbach, L. C. Hicks, M. Littmann, H. F. Shannon, G. W. Weiner.

Ferrous Metallurgy: C. W. Spencer, Chairman, H. I. Aaronson, G. S. Ansell, D. J. Blickwede, J. W. Cahn, A. J. Lena, G. R. Speich, F. E. Werner.

Heat Treating: R. L. Fullman, Chairman.

High Temperature Alloys: R. W. Fountain, Chairman, W. R. Clough, E. J. Dulis, E. S. Jones, D. W. Levenson, F. M. Richmond, Paul Shahinian, O. D. Sherby.

Joining: P. J. Rieppel, Chairman, F. R. Baysinger, W. D. Doty, C. E. Hartbower, George Hoppin, Kenneth Koopman, A. R. Lytle, E. F. Nippes, T. L. Oberle, G. R. Rothschild.

Melting and Casting: W. J. Childs, Chairman, Bruce Chalmers, M. C. Flemings, R. E. Grace, R. E. Leontis, H. W. Lownie, Jr., D. V. Ragone, L. W. Smith, W. A. Tiller, J. L. Walker.

Non-Ferrous Metallurgy: D. J. McPherson, Chairman, W. T. Bettis, J. T. Eash, J. W. Frederickson, F. H. Wilson.

Nuclear Metallurgy: W. D. Manly, Chairman, S. H. Bush, Premo Chiotti, H. H. Chiswik, B. W. Dunnington, J. A. Fellows, R. B. Gordon, A. N. Holden, R. T. Huntoon, C. E. Lacy, D. E. Thomas, M. J. Whitman.

Physical Metallurgy: P. G. Shewmon, Chairman, B. F. Brown, J. B. Clark, R. S. Davis, Paul Flinn, J. J. Gilman, N. A. Gjostein, C. D. Graham, Lawrence Himmel, H. C. Rogers, J. W. Spretnak, D. A. Thomas.

Powder Metallurgy: J. D. Shaw, Chairman, H. H. Hirsch, Past Chairman, A. D. Schwope, Vice Chairman, A. R. Poster, Secretary J. Gurland, J. A. Yoblin, Members-at-large.

Refractory Metals: L. P. Jahnke, Chairman, J. H. Bechtold, R. W. Fountain, N. J. Grant, E. L. Harmon, J. J. Harwood, R. I. Jaffee, J. Maltz, C. P. Mueller, R. C. Nelson, R. M. Parke, I. Perlmutter, Marion Semchyshen.

(Continued on next page)

Refractory Metal Conf. Will Be In Chicago April 12-13, 1962

The 1962 Refractory Metal Symposium has been scheduled for April 12 and 13 in Chicago's Morrison Hotel, it was announced recently by Joseph Maltz, of the IMD Refractory Metals Committee, and John V. Russell, Chairman of AIME's Chicago section. A tentative outline of conference activities calls for two full days of technical sessions on Thursday and Friday with a fellowship dinner on Thursday evening.

Others active in planning the conference are L. P. Jahnke, General Electric Co., Chairman. IMD Refractory Metals Committee; E. L. Harmon, Ford Motor Co., Publicity; Ike Perlmutter, Wright Air Development div., USAF, Program; and A. B. Michael, Fansteel Metallurgical Corp., and R. L. Van Thyne, Armour Research Foundation, Local Arrangements.

Fracture Conference Planning Committee Is Announced

The appointment of Prof. H. W. Paxton, Carnegie Institute of Technology, as chairman of a special IMD Committee to coordinate organization of an International Conference on Fracture was announced in August by J. H. Jackson, 1961 division chairman. The conference is tentatively scheduled to convene in the Seattle area during the week of August 20-24, 1962.

Selected to head the Program Committee for the gathering is Hans Brunner, Boeing Scientific Research Labs., who hopes to develop a broad range of approaches to the problem of fracture in crystalline solids; these are to include not only fundamental physical aspects but engineering considerations as well.

At a meeting of the Conference Committee on August 7, careful consideration was given to a number of rustic sites as potential locations for the meeting.

Also included in the membership of the Conference Committee are the following: John Rizley, Convair-A div. of General Dynamics Corp.; William M. Armstrong, University of British Columbia; Earl C. Roberts, University of Washington; Earl R. Parker, University of California; John J. Gilman, Brown University; A. J. Forty, University of Bristol; D. C. Drucker, Brown University; and Cyril S. Smith, University of Chicago.

Volume 10 Published In Conference Series; Others Are Scheduled

Volume 10 in The Metallurgical Society's conference series has recently been published by Interscience Publishers. Entitled **Columbium Metallurgy,** the book contains the proceedings of a conference sponsored by the Hudson-Mohawk section and The Metallurgical Society at Bolton Landing, N. Y., June 9-10, 1960.

Edited by D. L. Douglass and F. W. Kunz, the volume contains the 32 papers presented at the meeting. They are grouped into four sections: *Fabrication; Mechanical Properties; General Metallurgy;* and *Oxidation and Corrosion.* Containing 764 pages, the volume is priced at $26.00 (AIME members' price $20.80).

Refractory Metals and Alloys, Volume 11 in the conference series, has recently been published by Interscience. Edited by M. Semchyshen and J. J. Harwood, the volume contains the proceedings of the conference held in Detroit, May 25-26, 1960. The conference was sponsored by the Refractory Metals Committee of IMD, The Metallurgical Society and the Detroit section. The 22 papers included in the volume are presented under the headings; *General Metallurgy of Refractory Metals and Alloys; Refractory Metals and Alloys;* and *General Engineering of Refractory Metals.* This volume, containing 635 pages, is priced at $22.00 (AIME members' price $17.60).

Metallurgy of Elemental and Compound Semiconductors, Volume 12 in the Conference Series, is scheduled for publication by Interscience about September 19. This volume, edited by Ralph O. Grubel, contains the proceedings of the conference which was held in Boston August 29-31, 1960. The conference was sponsored by the Semiconductors Committee of IMD, The Metallurgical Society and the Boston section. The 30 papers contained in the proceedings are grouped under six headings. They are: *Germanium and Silicon for Esaki-Tunnel Diodes; Dendritic Growth of Germanium; III-V Compound Growth and Solubility Considerations; Other Semiconductor Materials;* and *Evaluation.* This 506-page illustrated volume is priced at $13.00 (AIME members' price $10.40).

Proceedings of the 1959 conference, **Properties of Elemental and Compound Semiconductors** were published as Volume 5 of the conference series. Priced at $8.50, copies are still available.

Bar and Allied Products will be Volume 13 of the conference series, and is scheduled for publication about October 1. Edited by E. W. Earhart and John W. Griffin, the book will contain the proceedings of the conference which was held in Pittsburgh, January 18, 1961. It was sponsored by the Mechanical Working Committee of ISD, The Metallurgical Society, and the Pittsburgh section. The book, containing approximately 150 pages, will be divided into two parts, *Surface Preparation and Rolling of Primary Products* and *New Developments in Processing and Testing.*

Registrants at any of the four conferences will receive a copy of the Proceedings of that conference as part of their registration fee.

Committee Officers

(Continued from page 649)

Semiconductors: H. E. Bridgers, Chairman, F. L. Vogel, Vice Chairman, G. E. Brock, Secretary, J. B. Schroeder, Treasurer, D. C. Jillson, Past Chairman, Joseph Duga, H. C. Gatos, R. O. Grubel, Pascal Levesque, J. O. McCaldin, S. A. Prussin, A. J. Rosenberg, F. D. Rosi, Alan Strauss, W. A. Tiller, J. H. Wernick, D. B. Wittry.

Shaping and Forming: W. A. Backofen, Chairman, R. P. Carreker, Jr., L. F. Coffin, T. S. Howald, W. T. Lankford, R. W. E. Laiter, E. J. Ripling, C. F. Schrader, E. G. Thomsen.

Structural Materials: J. H. Rizley, Chairman, G. M. Ault, R. W. Fann, Jr., H. Greenberg, H. Y. Hunsicker, R. R. Kennedy, H. P. Munger, C. W. Phillips, N. E. Promisel, E. E. Reynolds, A. O. Schaefer, W. V. Ward, Sumio Yakawa.

Titanium: T. E. Perry, Chairman, W. H. Sharp, Vice-Chairman, D. W. Levinson, Secretary, H. J. Boertzel, C. I. Bradford, G. L. Durfee, W. L. Finlay, R. L. Folkman, J. W. Fredrickson, Julian Glasser, R. I. Jaffee, T. W. Lippert, W. E. Lusby, Jr., I. Perlmutter, N. L. Reed, M. C. Sanz, David Swan, J. W. Sweet.

Vacuum Society Conf. Planned October 16-19 In Washington, D. C.

The Eighth Annual Symposium of the American Vacuum Society will be held in Washington, D. C. October 16-19, as part of the Second International Congress of the International Organization for Vacuum Science and Technology.

Of interest to metallurgists will be a special session on *Vacuum Metallurgy* consisting of 19 papers. Among the subjects to be discussed are fusion welding of beryllium, electron beam welding, the beam melting furnace, skull furnace operation, and induction furnaces.

Other sessions will cover *Ultra-High Vacuum, Vacuum Measurements Including Mass Spectrometry,* and *Evaporation and Thin Films.*

Pittsburgh Section's Off-the Record Meeting Scheduled November 3

The Pittsburgh section, AIME, will hold its annual Off-the-Record meeting on November 3, 1961 at the Penn-Sheraton Hotel. There will be five concurrent all-day sessions representing the following interests: Mineral Industries, Petroleum, Mining, Institute of Metals, and National Open Hearth.

The Mineral Industries program has been organized with the cooperation and assistance of the Office of Isotopes Development of the US Atomic Energy Commission (AEC). The theme of the program will be *Radioisotopes—New Horizons in the Mineral Industries.*

The morning session will begin with a welcoming address by Gerhard Derge, professor of metallurgical engineering at Carnegie Institute of Technology. The morning session will concern *What Radioisotopes Can Mean to the Minerals Industry,* and will include a paper on *Radioisotopes—Profitable New Tools for Industry* by Paul C. Aebersold of AEC. There will be a film on *Industrial Applications of Radioisotopes* provided by the AEC, followed by a paper on *Implications of Radioisotopes to the Minerals Industries.*

The second part of the morning session will concern *Radioisotopes in Geology,* and will include two papers.

The first part of the afternoon session will deal with *Opportunities in Industrial Radioisotope Technology.* The second part of the session will deal with *Radioisotope Applications in Mineral Processing,* and will feature a paper on *Application of Nuclear Density Gauges in the Mineral Industry,* by H. L. Cook, Jr. of the Ohmart Co.

There will also be papers on *Some Radiotracer Applications in Metallurgical Processing* by K. G. Broadhead and H. H. Heady of the US Bureau of Mines' Reno Metallurgy Research Center; *Radioisotopes for Coarse Ore Concentration* by Harald Ramdohr and Irene Donadieu of Massachusetts Institute of Technology; *Study of Radioisotope Applications in Copper Mining and Extraction—A Case History* by D. Bandel, Tracerlab, Inc.; and *Neutrons and Their Applications in the Mineral Industries* by M. M. Turkanis, Nuclear Materials & Equipment Corp.

Education

(Continued from page 611)

are provided by about two-thirds of the nation's engineering educational institutions. About half as many offer some courses at the graduate level.

A brochure describing the new program is available from the Dept. of Geology, College of Mines, University of Arizona, Tucson, Ariz.

Personals

(*Continued from page 609*)

Michael Tennenbaum, formerly assistant general manager of technical services at Inland Steel Co., has been appointed general manager of research and quality control.

Maurice L. Torti has been promoted to the post of director of metallurgical research for the Metals div. of National Research Corp. He joined the company in 1956.

Clifford E. Weber has been named director of fuels and materials at Atomics International, a division of North American Aviation, Inc. He joined the company in January after being associated for 15 years with General Electric's Knolls Atomic Power Laboratory.

Floyd S. "Dutch" Weimer, manager of the Great Falls reduction department of The Anaconda Co. retired July 1. **Lawrence J. Ingvalson,** general superintendent of the Great Falls plant, succeeded Mr. Weimer as manager, and **Leonard C. Powell,** formerly assistant general superintendent, became general superintendent.

T. B. Winkler has been promoted from div. head to director of research, processes, at Bethlehem Steel Co. At the same time, **D. J. Blickwede,** div. head, was named director of research, applications, and **J. W. Frame** was promoted to associate director, physical metallurgy.

New Student Members

University of British Columbia
McAndrew, Robert T.

Case Institute of Technology
Hesse, Arlan A.

Columbia University
Bernstein, Irving M.
Ohh, Sekyu

Missouri School of Mines
Kopaskie, Bernard E.

University of Pittsburgh
Clark, William G.

Universitat Technische (Germany)
Hamel, Hans, J., Berlin, Germany

Virginia Polytechnic Institute
Lovelace, William S.
(Graduated)
Kaloz, Arthur C., Cleveland, Ohio

Carnegie Institute of Technology
Haverstraw, Robert C.

Case Institute of Technology
Schwab, David E.

Drexel Institute of Technology
Bubar, Stanley F.

Massachusetts Institute of Technology
Berman, Herbert S.
Born, Richard D.
Heidekland, Herman R.

Purdue University
Hever, Charles F.
Krontz, Max M.

University of Queensland (Australia)
Grant, Roderick M.

Rensselaer Polytechnic Institute
Eisenbud, Leonard

Stanford University
Hinzer, Fritz
Zupp, Richard R.

Texas Western College
Rede, Hector

NECROLOGY

Year Elected	Name	Date of Death
1928	Adams, F. J.	Apr. 10, 1961
1942	Heide, Harold E.	June 20, 1961
1958	Henry, Russell P.	Oct. 7, 1960
1958	Keefe, Robert M.	June 17, 1961
1920	Kew, Wm. S. W.	June 4, 1961
1953	Pittman, E. M. Jr.	Nov. 19, 1960
1920	Rau, C. E. S.	Unknown
1940	Rostosky, A. Jr.	Aug. 3, 1960

Reginald Scott Dean

The death of Dr. Dean removes from the metallurgical world an outstanding scientist and engineer; and for those who were beneficiaries of his talents through the years by virtue of his consultation service, this removal reaches outstanding proportions.

He was born in Rolla, Mo., in 1897; his father, Professor George Dean was professor of mathematics at the Missouri School of Mines.

Dr. Dean graduated from the Missouri School of Mines and Metallurgy of the University of Missouri with a Bachelor's degree in 1915, followed by a Master's degree in 1916 and a professional degree in metallurgical engineering a few years later. His Ph.D. degree was earned at the University of Maryland in 1936.

Dr. Dean's professional activities were with the American Zinc Lead and Smelting Co.; the Anaconda Copper Co.; and as chief metallurgist of the Western Electric Co. for nine years. The US Bureau of Mines had his services for 13 years as chief engineer of the Metallurgical div., and as assistant director for four years ending in 1946. He was president of Chicago Development Corp., from 1946-1961, and a consulting engineer for a like period.

The author of two books (one on Theoretical Metallurgy in 1922, and one on Electrolytic Manganese in 1952), Dr. Dean held over 100 patents covering the fields of beneficiation, physical metallurgy, chemical processes for metal production, and electrochemistry.

In 1946 Dr. Dean established his own laboratory in Riverdale, Md., and became consultant for many companies. He conducted several research projects on batteries and titanium for the Army and Navy.

In addition to being a professional engineer, Dr. Dean was a registered patent agent, which gave him the privilege of acting as patent attorney for himself and others. Dr. Dean's most recent work included processes for production of pure chromium, titanium, and zirconium by molten electrofining.

MEMBERSHIP

Proposed for Membership
Metallurgical Society of AIME

Total AIME members on July 31, 1961, was 35,226; in addition 2,129 Student Members were enrolled.

ADMISSIONS COMMITTEE

W. L. Brytczuk, Chairman; S. C. Carapella, Jr.; J. W. Hanley; T. D. Jones; H. C. Larson; Harold Margolin; Shadburn Marshall.

The Institute desires to extend its privileges to every person to whom it can be of service, but does not desire as members persons who are unqualified. Institute members are urged to review this list as soon as possible and immediately to inform the Secretary's office if names of people are found who are known to be unqualified for AIME membership.

Members

Bickel, Joseph E. Jr., Syracuse, N. Y.
Borneman, Paul R., Brackenridge, Pa.
Chou, Ned. J., New York, N. Y.
Malone, Lloyd M., Fairfield, Ala.
Maltz, Joseph, Bethesda, Md.
Moxon, Arthur L., Brookfield, Ill.
Patty, Paul L., Florence, Ala.
Shapiro, Lester I., Wilkes Barre, Pa.
Sybicki, Edmund H., Kew Gardens, N. Y.
Yorke, David G., Pittsburgh, Pa.

Associate Members

Atkinson, Donald A., Bradford, Pa.
Maxwell, Robert M., New York, N. Y.
Petty, Carl L., Philadelphia, Pa.
Robinson, John A., Philadelphia, Pa.

Junior Members

Khanna, Sniv D., Bethlehem, Pa.
Krutenat, Richard C., Cromwell, Conn.
Spencer, Russell D., Detroit, Mich.

REINSTATEMENT—CHANGE OF STATUS

Associate to Member

Mitilineos, John D., Mount Vernon, N. Y.

Junior to Member

Turman, Robert E., Gary, Indiana

Student to Member

Salkovitz, Edward I., Forest Heights, Md.
Shepard, Lawrence A., Cambridge, Mass.
Trudeau, Guy J., Shawinigan, Que., Canada

SPECIAL REINSTATEMENT

To Junior

Skrill, Myron M., Chester, N. Y.
Gardner, John N., Salt Lake City, Utah

CHANGE OF STATUS

Associate to Member

Thomas, John G., West Chester, Pa.

Boston Section Elects Officers for 1961-62

The Boston section, AIME, has elected new officers and executive committee members for 1961-62. Larry Kaufman of Manufacturing Laboratories, Inc., Cambridge, Mass., is chairman, and Ernest P. Abrahamson, of Watertown Arsenal is vice chairman. Secretary-treasurer is S. Victor Radcliffe, Manufacturing Laboratories, Inc.

The members of the executive committee are R. S. Bray, Alloyd Corp.; Loring Frazier, Metal Hydrides, Inc.; Pascal Levesque, Electronic Metals and Alloys; and Robert A. Stauffer, National Research Corp.; Richard S. Davis, Arthur D. Little, Inc.; Thomas B. King, Massachusetts Institute of Technology; William C. Paynton, Metals and Controls, Inc.; and David A. Thomas, Massachusetts Institute of Technology.

Coming Events

Sept. 14-15, 1961, Joint Engineering Management Conference, Roosevelt Hotel, New York.

Sept. 17-20, 1961, Commemoration of the 50th Anniversary of Froth Flotation, Cosmopolitan Hotel and Brown Palace Hotel, Denver, Colo.

Sept. 22, 1961, Cleveland Section, NOHC, All-Day Technical Meeting, Sheraton-Cleveland Hotel, Cleveland, Ohio.

Oct. 2, 1961, Chicago Section, NOHC, Fall Technical Dinner Meeting, Phil Smidt's Restaurant, Hammond, Ind.

Oct. 2-3, 1961, 29th Annual Meeting, Engineers' Council for Professional Development, Sheraton Hotel, Louisville, Ky.

Oct. 5-6, 1961, Southwestern Section, NOHC, Annual Fall Meeting, President Hotel, Kansas City, Mo.

Oct. 16-18, 1961, Iron and Steel Institute (U.K.) international conference on The Metallurgy of Beryllium, Royal Commonwealth Society, London, W.C. 2.

Oct. 17-Nov. 7, 1961, Visit of the Iron & Steel Institute (United Kingdom) to the United States.

Oct. 20, 1961, Eastern Section, NOHC, 15th Annual Technical Conference, Warwick Hotel, Philadelphia.

Oct. 23-26, 1961, Fall Meeting of The Metallurgical Society of AIME, Pick-Fort Shelby Hotel, Detroit.

Nov. 1-3, 1961, Annual Pittsburgh Diffraction Conference, Mellon Institute, Pittsburgh, Pa.

Nov. 3, 1961, Pittsburgh Section, 10th Off-the-Record Meeting, Penn-Sheraton Hotel, Pittsburgh, Pa.

Nov. 13-16, 1961, Seventh Annual Conference on Magnetism and Magnetic Materials, Hotel Westward Ho, Phoenix, Ariz.

Nov. 14, 1961, Buffalo Section, NOHC, 12th Annual Meeting, Royal Connaught Hotel, Hamilton, Ont., Canada.

Nov. 29-30, 1961, Iron and Steel Institute (U.K.) Fall General Meeting on Future of Ironmaking in the Blast Furnace, London.

Dec. 4, 1961, Annual Meeting, Arizona Section, AIME, Pioneer Hotel, Tucson, Ariz.

Dec. 6-8, 1961, 19th Electric Furnace Conference, Penn-Sheraton Hotel, Pittsburgh, Pa.

Jan. 15-17, 1962, Minnesota Section, AIME, Annual Meeting held jointly with 23rd Annual Mining Symposium, Hotel Duluth, Duluth, Minn.

Jan. 17, 1962, AIME Mechanical Working Conference, Pick-Congress Hotel, Chicago.

Feb. 18-22, 1962, AIME Annual Meeting, New York, N. Y.

Apr. 9-11, 1962, National Open Hearth Steel Conference and Blast Furnace, Coke Oven, and Raw Materials Conference, Sheraton-Cadillac Hotel, Detroit.

Apr. 12-13, 1962, Chicago section, AIME, in cooperation with IMD Refractory Metals Committee, Refractory Metal Symposium, Morrison Hotel, Chicago, Ill.

Apr. 12-14, 1962, AIME Pacific Southwest Minerals Industry Conference, Palace Hotel, San Francisco, Calif.

April 26-28, 1962, AIME Pacific Northwest Metals and Minerals Conference, Ben Franklin Hotel, Seattle, Wash.

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THE METALLURGICAL SOCIETY OF AIME

1961 Fall Meeting Program

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EMD Program Committee

Contents

Calendar of Events	Page 5
Condensed Program	Page 2
Index of Authors and Chairmen	Page 43
Technical Sessions	Beginning on Page 6

REGISTRATION INFORMATION

Fall Meeting Registration Area — Mezzanine of Pick-Fort Shelby Hotel

Hours: Sunday, October 22 3:00-9.00 pm
Monday-Wednesday, October 23-25 8:00 am-4:00 pm
Thursday, October 26 8:00 am-3:00 pm

Registration fees:

AIME members (except student members)	$ 5.00
Nonmembers	$10.00
Nonmember Students	$ 1.00

Condensed Program - Fall Meeting of The Metallurgical Society
Pick-Fort Shelby Hotel, Detroit

Sunday, Oct. 22, 8:00 pm	Michigan Room Statler Hilton Hotel	Joint Session on Professional Activities		
	Terrace Room (Lower level)	**Ballroom (Mezzanine)**	**Shelby Room (Mezzanine)**	**Sky Room (21st Floor)**
Monday, Oct. 23, 8:00 am	Protective Coatings and Processing of Refractory Metals pp. 6-7	Theory of Work Hardening p. 7	Effects of Surface and Environment on Strength pp. 7-8	High Purity Iron and its Dilute Solid Solutions p. 8
Monday, Oct. 23, 2:00 pm	Processing of Refractory Metals pp. 8-9	Dislocations and Their Interactions pp. 9-11	Effects of Surface and Environment on Strength p. 11	High Purity Iron and its Dilute Solid Solutions p. 12
Tuesday, Oct. 24, 9:00 am	Titanium in 1975 p. 12	Continuous Casting (ISD) p. 12	Effects of Surface and Environment on Strength pp. 13-14	Physical Metallurgy of Refractory Metals pp. 14-15
Tuesday, Oct. 24, 2:00 pm	Titanium in 1975 p. 15	Working of Cast Structures (ISD) p. 15	Vacuum Techniques in Metallurgy (EMD) p. 15	Physical Metallurgy of Refractory Metals pp.16-17
Wednesday, Oct. 25, 9:00 am	Nuclear Fuel Reprocessing (EMD) pp. 17-18	Work Hardening and Strengthening Mechanisms pp. 18-20	Vacuum Techniques in Metallurgy (EMD) p. 20	Oxidation and Corrosion p. 21
Wednesday, Oct. 25, 2:00 pm	Nuclear Fuel Reprocessing (EMD) pp. 21-23	Diffusion pp. 23-24	Phase Relationships and Thermodynamics pp. 24-26	Deformation I pp. 26-28
Thursday, Oct. 26, 9:00 am	Deformation II pp. 28-30	Phase Transformations and Pressure Effects pp. 30-32	Surfaces pp. 32-33	Recrystallization, Grain Growth, and Textures pp. 33-35
Thursday, Oct. 26, 2:00 pm	Solidification pp. 35-37	Precipitation and Ageing pp. 37-39	Structure and Properties pp. 39-41	Powder Metallurgy and Processing of Metals pp. 41-42

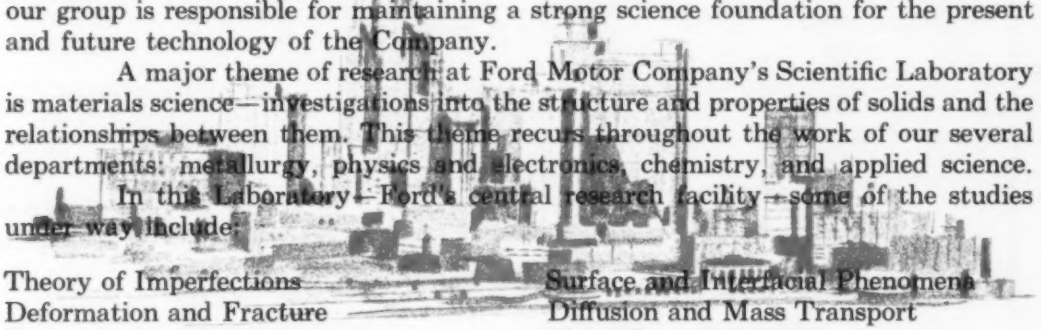
WELCOME TO DETROIT

We at the Ford Scientific Laboratory take great pleasure in welcoming you to the 1961 Meeting of the Metallurgical Society. We think you will like the bright new look of Detroit's skyline and the facilities of Cobo Hall. This year's meeting in Detroit holds particular significance to us, for it provides a fitting climax to the 10th anniversary celebration of the Ford Scientific Laboratory.

During this 10-year period, we have expanded from a small nucleus of people to nearly 300 persons—and we are still growing. Although this is a small fraction of the 10,000 technical people who make up the Ford Research and Engineering Center, our group is responsible for maintaining a strong science foundation for the present and future technology of the Company.

A major theme of research at Ford Motor Company's Scientific Laboratory is materials science—investigations into the structure and properties of solids and the relationships between them. This theme recurs throughout the work of our several departments: metallurgy, physics and electronics, chemistry, and applied science.

In this Laboratory—Ford's central research facility—some of the studies under way include:



Theory of Imperfections
Deformation and Fracture
Strengthening Mechanisms
Corrosion and Surface Chemistry
Surface and Interfacial Phenomena
Diffusion and Mass Transport
Phase Transformations
Sintering Mechanisms in Non-Metallics

The Scientific Laboratory enjoys the unique distinction of being the only industrial laboratory budgeted on a five-year basis. This budget program has assured the Laboratory's steady growth by authorizing a major increase in technical staff. It provides for continuity of basic and applied scientific research programs without regard to cyclical economic fluctuations.

The fundamental element in the Laboratory, however, is a core of creative, talented people who seek to gain the deepest possible understanding of the physical sciences.

We hope your visit to Detroit will be most pleasant and satisfying.

The Staff
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THE DETROIT SECTION
AND
THE METALLURGICAL SOCIETY OF AIME

REQUEST THE PLEASURE OF YOUR COMPANY AT THE FALL DINNER

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TUESDAY, OCTOBER 24 PICK-FORT SHELBY HOTEL
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4

CALENDAR OF EVENTS

Sunday, October 22

3:00 to 9:00 pm	Registration (Mezzanine)

Committee Meetings

4:00 pm	IMD Membership Committee (Georgian Room)
4:30 pm	MetSoc Committee on the Metallurgical Profession (Shelby Room)
6:30 pm	IMD Committee on Alloy Phases (Georgian Room)

Monday, October 23

8:00 am	Briefing for Authors and Chairmen (Cass Room)
8:00 am to 4:00 pm	Registration (Mezzanine)
12 noon	IMD Executive Committee Luncheon (River Room)

Committee Meetings

12:15 pm	IMD Chemistry and Physics of Metals Committee (Commerce Room)
4:00 pm	IMD Publications Committee (Commerce Room)
4:30 pm	MetSoc Education Committee (Georgian Room)
5:00 pm	IMD Refractory Metals Committee (Century Room)

Tuesday, October 24

8:00 am	Briefing for Authors and Chairmen (Cass Room)
8:00 am to 4:00 pm	Registration (Mezzanine)
8:00 am	MetSoc Board of Directors Breakfast (River Room)
12 noon	Yale Metallurgical Alumni Luncheon (Lafayette Room)
6:30 pm	Cocktail Party for holders of Fall Dinner tickets (Terrace Room)
7:00 pm	Fall Dinner (Terrace Room)

Committee Meetings

9:30 am	IMD Corrosion Resistant Metals Committee (Georgian Room)
1:30 pm	MetSoc Programs Committee (Georgian Room)
3:30 pm	ASTM Comm. B-2, Sub. VIII, Cb Task Group (Wayne Room)
	IMD Programs Committee (Commerce Room)
4:00 pm	IMD High-Temperature Alloys Committee (Century Room)
4:30 pm	IMD Physical Metallurgy Committee (Georgian Room)
4:30 pm	IMD Structural Materials Committee (Commerce Room)

Wednesday, October 25

8:00 am	Briefing for Authors and Chairmen (Cass Room)
8:00 am to 4:00 pm	Registration (Mezzanine)
9:00 am	AIME Board of Directors Meeting (River Room)
12 noon	AIME Board of Directors Luncheon (Fort Room)

Committee Meetings

9:00 am	ASTM Comm. B-2, Sub. VIII, Mo Task Group (Commerce Room)
2:00 pm	MetSoc Publications Committee (Commerce Room)
2:00 pm	IMD Nuclear Metallurgy Committee (Georgian Room)
2:00 pm	ASTM Comm. B-2, Sub. VIII, Tungsten Task Group (Wayne Room)
3:00 pm	IMD Non-Ferrous Metallurgy Committee (Century Room)
4:00 pm	IMD Melting and Casting Committee (Georgian Room)

Thursday, October 26

8:00 am	Briefing for Authors and Chairmen (Cass Room)
8:00 am to 3:00 pm	Registration (Mezzanine)
9:00 am	ASTM Comm. B-2, Sub. VIII, Thorium Task Group (Wayne Room)

SUNDAY, OCTOBER 22

Joint Session on Professional Activities

8:00 pm — Michigan Room, STATLER HILTON HOTEL

(Co-sponsored by MetSoc Committee on the Metallurgical Profession and ASM Development Committee)

Chairmen:

W. D. Manly, Oak Ridge National Laboratory, and *W. R. Hibbard, Jr.*, General Electric Co.

The Program of the Metallurgy and Ceramics Foundation: *G. A. Roberts*, Vanadium Alloys Steel Corp.

The Question of Establishing a National Academy of Engineering: *H. K. Work*, New York University.

MONDAY, OCTOBER 23

Protective Coatings and Processing of Refractory Metals

9:00 am to 12:00 m — Terrace Room

(Sponsored by IMD Refractory Metals Committee)

Chairmen:

E. D. Sayre, General Electric Co., and *O. O. Srp*, Wright Aeronautical Development Division.

Protective Coatings for Tantalum and Tantalum Alloys: *W. D. Klopp* and *H. R. Ogden*, Battelle Memorial Institute.

Protective coatings of aluminum, chromium, hafnium, silicon, titanium, binary combinations of these metals, zinc, and Al_2O_3 were applied to tantalum and evaluated for static oxidation behavior in the temperature range 2200° to 2700°F. The coatings were applied by flame spraying, hot dipping, and pack cementation. The latter process proved most attractive from the standpoint of coating uniformity, versatility with regard to coating composition, and ease of application. Process variables studied included pack composition, pack size, and deposition temperature.

Silicon and silicon-rich alloy coatings applied by pack cementation protected tantalum and a high-strength tantalum alloy from oxidation at 2700°F for at least 8 hr. Silicon-aluminum alloy coatings were equally as protective and appear to possess limited self-healing properties. Aluminum coatings also appear protective for unalloyed tantalum to at least 2700°F, but chromium, hafnium, and titanium coatings do not appear useful above 2200°F.

The Development of Oxidation Resistant Coating for Molybdenum: *Pao Jen Chao* and *D. K. Priest*, The Pfaudler Co.

Prominent high-temperature mechanical properties of molybdenum-base alloys meet the requirements of aerospace applications in the temperature range of 2000° to 3000°F. Their potential at these temperature environments, however, is limited by complete lack of oxidation resistance. Protective coatings have been developed for molybdenum-base alloys, with the most advantageous coating technique being the pack diffusion process. Known techniques do not produce the required coating integrity, and the present research effort was undertaken to develop a desirable and reproducible coating system. An extensive screening study of various pack diffusion coating systems was made using three basic process concepts. Promising systems were evaluated in detail. Reliability and reproducibility were incorporated into the best coating through a statistical study of the variables affecting the coating process. Evaluation data for two promising coatings are presented. Scale up operation, suitability of methods of fabrication of substrates, nondestructive tests, and repairability are being investigated.

An Oxidation Resistant Aluminide Coating on Columbium Alloy F-48: *Stephen Foldes*, General Electric Co.

Three modifications of an aluminide coating for short time oxidation protection at 2500°F were studied. The aluminide coating was supplied by dipping in an organic slurry of Al-10 pct Si alloy, re-dipping in a slurry of aluminum powder, and firing. The three modifications studied were: 1) electroplating the columbium alloy with silver, 2) enriching the surface with titanium by diffusion, and 3) by incorporating silver in the Al-11 pct Si alloy.

In still air the silver electroplate-aluminide coating combination protected F-48 for 10 hr at 2500°F. Enriching the surface with titanium before applying the aluminide coating gave 2 hr protection under the same conditions. The Al-Ag-Si slurry provided less than ½ hr protection. Only the silver plate—aluminide coated F-48—did not exhibit a marked decrease in its room temperature ductility after oxidation.

The oxidation resistance of selected Cb-Al alloys was also cursorily surveyed. Increasing the aluminum content improved the oxidation resistance but reduced ductility. Additions of Ti + Cr and Ti + Si decreased the Al content required for an equivalent oxidation resistance in alloys devoid of Ti, with a concomitant improvement of ductility of the compounds. Identification of phases existing in an oxidation resistant, aluminide coating on a Cb-25 pct Ti alloy were made by X-ray diffraction in order to synthesize similar constituents on the surface of F-48 panels.

Detailed studies were carried out on silver plate-aluminide coatings. On oxidation at 2500°F. Cb Al_2 appeared at the coating-metal interface. The main constituent of the coating was established by X-ray diffraction as Cb Al_3. The phase identification work has been supplemented by electron microscopy and X-ray microemission analyses to elucidate a possible mechanism in the prevention of notch embrittlement of the base alloy by silver plating before application of the aluminum slurries. Although there was no evidence of a distinct silver-rich phase at the coating-metal interface in the as applied or oxidized condition, concentration of Ag and Si existed in the coating close to the Cb Al_3 interfacial layer. This supported the speculation that the presence of silver rich areas could be contributory in the prevention of the propagation of cracks from the outer coating into the base metal.

The excellent oxidation resistance and the ability of the coating to cover fairly sharp corners are major considerations in the usefulness of this coating for applications where the plating step is the only limiting factor.

Properties of the Refractory Metals Sprayed Under Controlled Environment: *M. A. Levinstein*, General Electric Co.

Work performed under a WADD contract on W, Mo, Ta, and Cb is covered in this report. The equipment set-up for spraying under controlled environment condition is described. Sample preparation and configuration are discussed. Mechanical properties up to 4500°F are reported. Microstructures of the deposits are analyzed. Effects of alloying additions are also reported.

Some Observations on the Infiltration of Porous Structures of Molybdenum, Tungsten and Their Carbides with Copper in Vacuum and in Hydrogen: *H. H. Hirsch*, General Electric Co.

Pressed bodies of Mo, W, MoC, and WC were infiltrated with zone refined and OFHC copper. The greatest emphasis was placed on W-Cu composites. Infiltrations were carried out using vacuum, hydrogen, and a combination of these for the infiltration atmospheres.

The thoroughness of infiltration is influenced by the atmosphere, heat treatment, and supporting material. The total gas content of the infiltrated bodies were obtained and related to the atmosphere, heat treatment, and type of copper. A recommended procedure is given for good infiltrations with low gas contents.

A Study of Brazing Alloy Systems for Joining Columbium Base Alloys: *W. R. Young* and *E. S. Jones*, General Electric Co.

Increasing attention is being focused on the use of Cb-base alloys for a variety of nuclear, aerospace, and other high temperature applications. Several laboratories have identified promising alloy compositions, and the Dept. of Defense is sponsoring a num-

MONDAY, OCTOBER 23

ber of programs to develop the technology of producing these alloys in quantity.

In order to be used in the manufacture of components, it is desirable to be able to join Cb alloys by brazing. The present study, sponsored by WADD Materials Central, was undertaken with the objective of identifying promising braze alloy systems for joining of two types of Cb alloys. One base alloy, Cb + 1Zr, was chosen as being representative of the low strength, easily fabricable alloys, while F-48 alloy (Cb + 15W + 5Mo + 1Zr) was selected as representative of the high strength, more difficult to fabricate alloys.

The criteria for selecting braze alloy systems for study were: 1) melting range and 2) compatibility with the Cb base alloys. For this study, compatibility is defined as absence of intermediate and low melting eutectic phases between the braze and Cb-base alloy.

Since the expected applications of these Cb alloys involved two temperature regimes—to 2500°F for F-48 and 1800° to 2200°F for Cb + 1Zr—brazing alloy systems were selected with melting ranges of potential usefulness for each temperature range. A literature survey indicated several binary alloy systems based on Ti, Zr, V, Ta, and Cb itself which exhibited appropriate melting temperatures. The selected brazing alloys were arc melted preparatory to brazing studies. Melting point determinations were made, and the flow and diffusion characteristics on the two base metals were observed.

Alloys with attractive melting, wettability, and diffusion characteristics were additionally studied for long time diffusion characteristics, and braze joint strength. The effect of braze cycle on parent metal hardness and microstructure, and the bend transition temperature of brazed panels were also determined.

As a result of this study, braze alloy systems were identified which show promise for use in joining Cb alloys for service in the 2000° to 2500°F range. Ternary alloys are suggested as having even more attractive possibilities.

Attendance at sessions limited to persons registered for Metallurgical Society Fall Meeting. Registration information—Page 2.

Selected Topics in the Theory of Work Hardening

9:00 am to 12:00 m — **Ballroom**

(Sponsored by IMD Chemistry and Physics of Metals Committee)

Chairman:

G. M. Pound, Carnegie Institute of Technology.

Presentations by *Z. S. Basinski*, National Research Council (Canada), *Jens Lothe* and *J. P. Hirth*, Carnegie Institute of Technology, and others.

Symposium on the Effects of Surface and Environment on Strength I

9:00 am to 12:00 m — **Shelby Room**

(Sponsored by the Committees on Corrosion Resistant Metals and Chemistry and Physics of Metals)

Chairman:

F. N. Rhines, University of Florida.

The Interaction of Surface and Strength Phenomena: *E. S. Machlin*, Columbia University.

The state of surface may affect flow and fracture strengths in the following ways: 1) by influencing the rate of escape of dislocations out through the surface; 2) by influencing the motion of dislocations that intersect the surface; 3) by affecting the energy of newly-formed surfaces at crack spearheads; and 4) by alteration of the elastic constants for a sufficient distance in from the surface to affect dislocation generation. Examples of these effects are cited. More importantly, the areas where insufficient precise experimentation exists are listed and suggestions are made for future research.

Recent British Thin Foil Experiments: *John Silcox*, Cambridge University.

Current British work using the thin foil microscopy technique will be reported. The contrast left behind by dislocations when they move can be interpreted in terms of dislocations (with image dislocations) left behind at the surface of the thin foil. It is also possible to utilize the slip trace contrast to study the rearrangement of dislocations during thinning. These and other topics will be discussed.

Stresses in Overgrown Films on Crystalline Substrates: *N. Cabrera* and *J. H. van der Merwe*, University of Virginia.

The energies connected with an interface between a crystalline film and a crystalline substrate of a different substance for the simple case that the interfacial spacings differ in one direction only are calculated. The interfacial contact can in this case be analysed in terms of a row of edge dislocations with Burgers vectors in the interface. The energies are calculated in terms of film thickness h, interfacial misfit, interfacial bonding, and relative hardness of film and substrate. The interfacial bonding as defined here includes tangential periodic forces of the Peierls-Nabarro type to account for the periodicity of the lattices and normal forces induced by the former. The interfacial energy converges rapidly with h to a limiting value corresponding to a film with infinite thickness—the limiting value being practically attained when h matches half the dislocation spacing p.

The atomic spacing of the film in the lowest energy state of the system differs from the bulk spacing. The two main reasons are that the film is in a different force field even when the bulk atomic spacings of film and substrate are the same and that energy is associated with the interfacial dislocations. The former is important when h is less than or equal to the range of atomic forces. The presence of the interfacial dislocations requires the film to be homogeneously strained in the lowest energy state. The calculations show that below a critical value of misfit, the appropriate strain corresponds to exact fit of film and substrate whereas beyond this, the required strain is an order of magnitude lower than the critical misfit. Both values depend on the parameters mentioned above. The critical value can be of the order of 10 pct for a soft monatomic film which is strongly bound to the substrate while both values are roughly inversely proportional to the film thickness. When the conditions are such that equilibrium is not attained at each growth stage mestastable states may result which finally lead to plastic yield of the film. The dislocations nucleated in this manner are unlikely to generate slip in the substrate. Energy considerations also show that for an unstrained film the stable position of the dislocation array is at the interface independent of bonding strength and relative hardness. This may not be the case when either the film is strained or the boundary is of a more general type which also contains dislocations with Burgers vectors inclined to the interface. At least one case is conceivable where a film may force an interfacial dislocation into the substrate. The influence of the foregoing, and other surface stresses, on the mechanical properties of the substrate will be discussed.

Supported by the US Atomic Energy Commission Contract AT-(40-1)-2488, code 4163.

An Electromechanical Effect in Semiconductors: *J. H. Westbrook*, General Electric Company, and *J. J. Gilman*, Brown University.

The resistance of semiconducting crystals to indentation deformation has been found to be lowered significantly (up to 60 pct) by the simultaneous presence of a small potential (0.05 to 10 v) between the indenter and the crystal surface. This electromechanical effect appears to be confined to the plastic strain region of a surface layer of material 2 to 3 microns deep.

A large body of experiments of varied nature have established that the effect is real and not spurious. Similar effects are observed in a number of different semiconductors (Ge, Si, InSb, and SiC) but not in metals or ionic crystals. The effect disappears at elevated temperatures but becomes enhanced below room temperature. It is not sensitive to the type of current carrier. The role of surface preparation, crystallographic orientation, kind and concentration of charge carrier, geometry, and time have also been examined. Other and possibly related experiments show significant enhancement of the surface photovoltage (the Dember potential) by a longitudinal electric field. All of these experiments are extensively documented and discussed, but no satisfactory model or mechanism has yet been conceived.

MONDAY, OCTOBER 23

Influence of Thermal-Mechanical History on the Embrittlement of Aluminum Alloys by Mercury: *H. Nichols* and *W. Rostoker*, Armour Research Foundation.

Liquid mercury embrittles all but unalloyed aluminum to some degree. Certain high-strength alloys are so embrittled that failure occurs at less than 20 pct of the yield stress. It is shown that intrinsic strength of the alloy itself is not the dominant factor. Pure aluminum, although restrained from yielding to very high stress levels as in Alclad, is not measurably embrittled. The 2024 alloy in the overaged state does not become more embrittled even though cold-rolled 95 pct to strength levels as high as the aged hardened state. Simple aging produces a trend with aging time of failure (wetted with Hg) above the yield to below the yield. The most severely embrittled state is the fully-aged condition with the superposition of a small amount of cold work. Some recovery of strength with large amounts of cold work is noted. These phenomena are discussed in terms of a model of the lowering of the surface energy for fracture by the circumstance of wetting the surface of aluminum with mercury. This work is supported by the Office of Chief of Ordnance, Dept. of the Army and is supervised by Frankford Arsenal.

High Purity Iron and Its Dilute Solid Solutions

9:00 am to 12:00 m — Sky Room

(Sponsored by IMD Ferrous Metallurgy Committee)

Chairman:

C. L. McCabe, Carnegie Institute of Technology.

Preparation and Analysis of High Purity Iron and Its Dilute Solid Soutions: *R. L. Smith, B. Park*, and *J. A. Van Westenberg*, Michigan College of Mining and Technology.

On the Iron Phase Transformation at High Pressures: *R. S. Davis*, Arthur D. Little, Inc.

Thermal, Magnetic, and Electrical Properties of Fe with Various Solutes: *Anthony Arrott* and *J. E. Noakes*, Ford Motor Co.

Processing of Refractory Metals

2:00 to 5:00 pm — Terrace Room

(Sponsored by IMD Refractory Metals Committee)

Chairmen:

W. H. Rostoker, Armour Research Foundation, and *Verne Pulsifer*, Fansteel Metallurgical Corp.

Processing and Properties of Arc Cast Tungsten Sheet: *C. P. Mueller* and *W. J. Schoenfeld*, Universal-Cyclops Steel Corp.

Conversion techniques for producing high quality arc-cast tungsten sheet in large sizes and thin gages have been accomplished The processing problems have been evaluated and a realistic approach has been taken to manufacture the desired product. Recrystallization data as well as mechanical property data have been generated for various sheet thicknesses. Present size limitations and possible solutions have been evaluated, and a method for producing 36 x 96 in. sheet is presented.

Arc Melting and Hot Workability of Tungsten Alloys: *R. G. Carlson*, General Electric Co.

This report contains the results of studies on the hot workability of tungsten alloys containing titanium, zirconium, silicon, aluminum, tantalum, carbon, and boron that were prepared by pressing the powders into electrodes, arc-melting these electrodes into ingots, and working the ingots into sheet.

From the results of these studies it was found that the hardness of tungsten is not greatly affected by any alloying addition except zirconium: 0.5 at pct zirconium addition reduces the as-cast hardness, ordinarily for unalloyed tungsten of ca. 64 to 58.4 Rockwell A. It was further found that boron, carbon, silicon, and zirconium, in decreasing order in the ranges investigated promote grain refinement in the ingots. From this work, it was also ascertained that the solubility of boron in tungsten was between 5 to 10 ppm.

The hot workability of tungsten could be increased by the addition of carbon and especially boron.

In a cursory investigation of the recrystallization temperature of the rolled material it was determined that it was increased 150°C by the addition of 0.5 at pct (0.029 wt pct) B, while the carbon additions apparently did not greatly increase the recrystallization temperature of 1250°C at the 50 pct recrystallization level for undoped tungsten sheet.

Evaluation of Variables Related to the Production of Arc Cast Molybdenum Alloy Sheet: *W. A. McNeish*, Universal-Cyclops Steel Corp.

This paper represents a summary of the work performed under Bureau of Naval Weapons Contract NOas 59-6142-c. A comprehensive study was conducted to determine the effects of consolidation practice and primary fabrication on the quality of sheet bar and subsequent sheet product. Variations in ingot diameter and single vs double melting were studied. Sheet bar fabrication practices included direct forging, direct extrusion, and combinations of these operations. Direct forging experiments were conducted in an argon atmosphere (InFab). Rolling experiments included all those variables which experience had shown to result in undesirable sheet properties. Sheet properties obtained for Mo + 0.5 pct Ti and Mo + 0.5 pct Ti + 0.1 pct Zr were fully analyzed.

Processing and Properties of Molybdenum Mill Products Produced from Massive Isostatically Pressed and Sintered Billets: *L. L. France* and *F. P. Giannakos*, Universal-Cyclops Steel Corp.

A brief description of the molybdenum powder isostatic pressing and sintering techniques used to produce the starting billet is presented. Resultant billet properties and inspection techniques are described. Billet size, in the case of sheet bar, was 2 in. x 10 in. x 24 in. and, in the case of round billet for bar production, the size was 4½ in. diam by either 24 or 48 in. long. Processing procedures are described for the production of various gage sheet —the maximum size being 30 in. wide by 96 in. long. In the case of bar processes, data is presented for final bar sizes of 2 in. diam and smaller. The resulting as-rolled and stress-relieved tensile and bend properties are presented. A comparison between sheet and bar produced by powder metallurgy techniques vs arc-cast techniques is made.

The Effect of Microstructure on the Workability and Properties of Vacuum Hot Pressed Columbium Alloy: *T. L. Robertshaw*, and *R. G. Frank*, General Electric Co., and *I. Perlmutter*, Wright Air Development Division.

Prealloyed powder of F-48 composition (15W-5Mo-1Zr balance Cb) was vacuum hot-pressed under various processing cycles. Although finer powders, higher temperatures, and longer times yielded fully dense billets of fine grain size, microstructural changes occurring during compaction inhibited ductility. Deleterious grain boundary precipitates, formed at the higher temperatures and longer times, could be traced to interstitial content of powder charges. Rollability, and hence ductility, was shown to be enhanced by compaction of pure powders above a threshold density and at temperatures below 3700°F.

Mechanical properties are compared with those obtained from two other consolidation techniques.

Techniques for the Preparation of High Purity Refractory Metals: *E. Fenn, M. Badiali*, and *R. Bakish*, The Alloyd Corp.

The preparation of high purity Mo, W, and Ta by distillation, sublimation and zone refining of the halides prior to hydrogen reduction is presented. Equipment used, processing variables, and experimental results on these high purity metals are discussed. A correlation between analytical data and processing variables is attempted.

MONDAY, OCTOBER 23

Strength of Sintered Nickel-Tungsten: *J. H. Brophy, H. W. Hayden,* and *J. Wulff,* Massachusetts Institute of Technology.

The transverse rupture strength of sintered compacts of tungsten containing small amounts of nickel has been studied as a function of mode of powder preparation, density, and nickel content. Sintering times and temperatures were in the range of 1 to 16 hr at 1000° and 1100°C. Strength was found to increase in specimens containing 0.25 pct Ni as density increased during the initial stage of the carrier phase sintering process proposed by the authors previously. Upon the onset of grain growth in the final stage strength was found to decrease. With increasing nickel contents up to 4 pct, strength increased, presumably due to the restriction of grain growth which was observed in these cases. Maximum strengths were observed at 73,000 psi in three-point loading. This was comparable to that of massive tungsten recrystallized in the presence of nickel and tested in the same way. The results were sensitive to the mode of powder treatment and ductility was negligible in all cases.

Dislocations and Their Interactions

2:00 to 5:00 pm — Ballroom

(Based on Submission of Research Abstracts)

Chairmen:

T. L. Johnston, Ford Motor Co., and *Norman Brown,* University of Pennsylvania.

Fast Moving Edge Dislocations on the (110) Plane in Anisotropic B.C.C. Crystals: *J. Weertman,* Northwestern University.

The elastic displacement field around a uniformly moving edge dislocation has been obtained for the case where the dislocation glides on a (110) plane in a <111> direction in an anisotropic bcc crystal. A general solution is found which involves elastic displacements in all three coordinate directions. The situation where the degree of anisotropy is small is analysed in detail. An equation is obtained for the velocity (the Rayleigh wave velocity) at which the shear stress on the slip plane of a moving dislocation is zero. Dislocations of like sign moving at velocities faster than this velocity attract rather than repel each other. It is concluded that when anisotropy is small and the elastic constant c_{11} is smaller than $c_{12}+2c_{44}$ (the more commonly occurring relationship between the elastic constants) the Rayleigh wave velocity is increased above its value in an isotropic crystal and therefore the extent of the velocity region where dislocations exhibit an anomalous behavior is decreased.

Sponsored by Air Force Special Weapons Center, Air Research and Development Command, Kirkland Air Force Base.

Fast Moving Edge Dislocations in Alpha Iron: *J. Cotner* and *J. Weertman,* Northwestern University

A calculation is made of the shear stress on the slip plane of an edge dislocation moving uniformly in the alpha iron lattice. The dislocation is taken to be on a (110) plane and moving in a <111> direction. It is found that the shear stress approaches zero at the Rayleigh wave velocity, 0.92c, where c is the shear velocity in the <111> direction. The limiting velocity of dislocation motion is 0.94c. In the velocity range between 0.92c and 0.94c dislocations of like sign attract one another. The extent of this velocity region in which dislocations exhibit anomalous behavior is three times smaller than the corresponding region in isotropic crystals. Hence, as compared with the isotropic case, it is more difficult to bring dislocations moving in iron on a (110) plane into a velocity region where coalescence of fast moving dislocations can occur. (It should be noted that this coalescence may lead to fracture.)

On the Nature of Dislocations Which Cause Nonbasal Slip in H.C.P. Metals: *H. S. Rosenbaum* and *M. L. Kronberg,* General Electric Co.

While cadmium, zinc, and magnesium crystals slip mainly on the basal plane, metals with lower c/a ratios, e.g., titanium, zirconium, and beryllium, slip mainly on nonbasal planes. Furthermore, even cadmium, zinc and magnesium crystals can be made to slip on nonbasal planes if they are suitably stressed. This report is concerned with the geometric nature of the dislocations which cause nonbasal slip; a hard sphere model is used to describe the atom movements required to produce slip.

Cadmium and zinc are known to slip on the system $\{11\bar{2}2\}\langle\bar{1}\bar{1}23\rangle$ at low temperatures. Using the hard sphere model and the assumption that atom movements preserve the largest number of nearest neighbor bonds, certain inferences are drawn regarding these dislocations. It is shown that a total dislocation with Burgers vector $b = 1/3\ \langle\bar{1}\bar{1}23\rangle$ can dissociate into partials whose Burgers vectors are also parallel to $\langle\bar{1}\bar{1}23\rangle$. The resulting stacking fault involves the breaking of one nearest neighbor bond for each atom in the fault. A similar analysis is used for the dislocations of the slip system $\{10\bar{1}1\}\langle\bar{1}\bar{1}23\rangle$.

The mechanism for slip in $\langle\bar{1}\bar{1}23\rangle$ directions suggested in this model is similar in concept to the *zonal dislocation* previously introduced by Kronberg to explain slip in β-uranium. That is, the dislocations involve a coherent transition zone that occupies a finite volume of the material.

Finally, slip on nonbasal planes containing the close-packed direction $\{10\bar{1}\ n\}\langle\bar{1}2\bar{1}0\rangle$ is considered. Slip on these systems does not require faults with transition zones.

Solution Hardening: *R. L. Fleischer,* General Electric Co.

Solution hardening is considered for the case of elastic interaction between a dislocation and a random solid solution. It is pointed out that the theory of Mott and Nabarro, which concerns the effect of misfit due to atomic size differences, includes only interactions due to the dilational portion of the strain field and therefore implies that only edge dislocations will be opposed; hence, screw dislocations may move freely. In addition, the strengthening of solid solutions has not been observed to be simply proportional to the fractional change in lattice parameter with composition $\epsilon_b \left(= \frac{1}{b}\frac{db}{dc} \right)$ as the theory suggests.

It is proposed that the major elastic interaction of a dislocation with a solute atom is due to the atom effectively having a shear modulus which differs from that of the matrix, so that individual atoms may be represented by local elastic hard or soft spots. It is shown that for edge dislocations this effect is comparable with that due to atomic size misfit. In addition, since the modulus effect provides an interaction with the shear portion of the field of a dislocation, screw as well as edge dislocations will be opposed.

In order to test experimentally predictions based on the modulus effect, it is necessary to have measurements of the fractional change in shear modulus with solute concentration $\epsilon_G \left(= \frac{1}{G}\frac{dG}{dc} \right)$. For copper alloys the calculation that the increase in flow stress due to alloying should be proportional to $\epsilon_G/1 + \epsilon_G/2$ appears to be in satisfactory agreement with existing data; for silver alloys there is little difference in the extent to which the predictions of the modulus and size effects are obeyed.

It is also noted that impurity pinning of all dislocations (not merely edge dislocations) will arise from modulus differences, so that yield points are to be expected.

Formation of Point Defects During Deformation: *H. K. Birnbaum,* University of Chicago.

The generation of point defects during plastic deformation and their subsequent interaction with dislocations has been studied in high purity copper. The techniques used were low temperature strain aging and elastic modulus measurements in the temperature range 77° to 373°K. Two types of point defects have been observed to anneal in the above temperature interval. Both defects interacted with dislocations to give rise to a small yield effect ($\Delta\sigma$) and an increase in the elastic modulus (M). At each aging temperature, two maxima in the values of $\Delta\sigma$ and M were observed as the aging time was increased. The characteristics of the yield effect indicated that point defect pinning of the dislocations occurred during aging. Analysis of the aging kinetics indicated that the data was consistent with a model of the point defect annealing in which the defects diffused to dislocation lines and then diffused along the dislocations to sinks. A brief description of this model will be presented. Using this model, values for the diffusion activation energies of the defects in the lattice and along the dislocations were derived. Analysis of the data indicated that the two defects were the interstitial and the vacancy.

MONDAY, OCTOBER 23

Density and Distribution of Dislocations in Copper Crystals Deformed in Tension: *J. D. Livingston*, General Electric Co.

Single crystals of 99.999 pct Cu have been deformed in tension. Four different tensile axis orientations have been studied: one single slip orientation, one double slip orientation ([211]), and two multiple slip orientations ([111] and [100]). Deformation was occasionally interrupted and the disclocation density measured using etching techniques capable of revealing dislocations intersecting (111), (110), or (100) faces of the crystals. The range of useful measurement was extended to high etch pit densities with the use of replicas for observation in the electron microscope. The variation with stress and strain of the average dislocation density so measured will be reported.

The distribution of dislocations was also found interesting and afforded information on dislocation sources and dislocation barriers. The (111) etch is capable of distinguishing the sign of individual edge dislocations, and thus for the crystals capable of study with the (111) etch, interesting additional information on the distribution of positive and negative dislocations has been obtained.

The relation of these various observations to current theories of deformation and work-hardening of fcc metal crystals will be discussed.

X-Ray Diffraction Micrographic Observations Dealing With the Nature and Origin of Dislocations in 'Pure' Zinc Monocrystals Solidified From the Melt: *R. W. Armstrong* and *J. M. Schultz*, Westinghouse Electric Corp.

Randomly oriented and specially oriented zinc monocrystals have been grown from the melt by the Bridgman technique and examined with the Berg-Barrett X-ray technique to obtain evidence principally on the nature and properties of the initial distribution of dislocations which exist within the solid. Our study deals with: 1) the characteristics of dense patterns of dislocation lines grown into the crystals and 2) the observation of dislocation rings, spiral loops, and slip dislocations. Evidence from this study indicates that an individual line of the X-ray fine structure may represent, in some cases, an individual dislocation but, in numerous other cases, an individual line represents a small group of parallel dislocations.

Dense patterns of long, straight dislocation lines lying in the 0001 (basal) planes have been observed. The presence of these dislocations in the crystal matrix after growth from the melt is not attributed to the dislocation formation mechanisms of impurity-cell formation or dislocation striation formation. The dislocation lines and the patterns which they form exhibit the following characteristics:

1) The arrays of lines appear to be independent of the crystal growth direction;

2) The arrays are composed of sets of roughly parallel lines running in definite directions in a given region of the crystal but a predominance of one direction over the others varies from region to region; and

3) The lines show considerable fine branching at high resolution; this observation indicates that the lines are often not individual dislocations and this conclusion is verified by the results of etching experiments.

Dislocation rings and spiral loops have been observed in the 0001 (basal) plane of the crystals. The loops and rings are divided into three groups according to their size and other characteristics:

1) Rings and spiral loops approximately 300 · microns diam are observed to closely resemble Frank-Read sources. These rings and loops have been definitely associated, in some cases, with inclusions (as suggested by Damiano). The loops are not affected by substantial crystal deformation.

2) Large dislocation rings approximately 1000 microns diam are observed, and they are associated with *polygonization* of dislocations. The ring centers appear to present substantial obstacles to the motion of slip dislocations.

3) Very small dislocation rings, less than 10 microns diam are observed near the limit of X-ray resolution. They may form by some mechanism other than inclusion association.

Groups of long, bowed dislocation lines have been caused to form by deformation near the crystal edge. The configuration of these groups is similar to the configuration of dislocation lines near subgrain boundaries.

Application of the Berg-Barrett X-Ray Technique to 'Seeing' Dislocations in Zinc: *J. M. Schultz* and *R. W. Armstrong*, Westinghouse Electric Corp.

An X-ray diffraction micrographic study using characteristic iron and chromium radiation has been made on zinc monocrystals grown from the melt by the Bridgman technique. The fine line structure of the X-ray diffraction spots has been associated with the intrinsic character and properties of individual or small groups of parallel dislocations rather than with anomalous X-ray effects, impurity effects, or crystal surface effects from the following evidence and considerations:

1) The individual lines and fine structure are observed in pure (99.999 wt pct) and zone refined material with variously prepared crystal surfaces;

2) Optical and interference microscopy allow us to differentiate between the crystal surface structure and the X-ray line structure;

3) The diffraction properties of the lines observed in the X-ray patterns are consistent with the properties of dislocations;
   a) the effects of X-ray divergence, the crystal specimen-X-ray film distance, and K_α splitting have been studied,
   b) the (planes and) Brugers vectors of the dislocations have been investigated;

4) The special configurations and general appearance of the lines are compared with the observations of dislocations in other crystals and with the appearance of low angle boundaries (to the extent of resolving individual dislocations in the boundaries);

5) The line networks yield random dislocation densities $\geq 10^5$ to 10^6 lines per sq cm and these measurements have been correlated with estimates from the Lambot technique and an interferometric study of bent specimens;

6) Deformation affects the X-ray fine structure;
   a) Scratches put into crystal surfaces change the line patterns,
   b) bending crystals produces new lines,
   c) mechanical twins produce additional lines in the crystal matrix; and

7) Chemical polishing and etching experiments combined with X-ray micrography show the crystal depth to which the reflected X-rays have penetrated, and demonstrate that the internal dislocation structure is revealed; dislocations are shown at the etch pits which occur ahead of advancing mechanical twins.

Dislocation Mobility in Magnesium: *G. S. Ansell, M. J. Roblin*, and *R. S. Goodrich*, Rensselaer Polytechnic Institute.

Stress-velocity measurements for both edge and screw dislocations in magnesium have been obtained by transmission electron microscopy by utilizing sequential photography. Edge dislocation velocities were measured on the basal glide plane and were found to vary from 3×10^{-4} to 8×10^{-7} cm per sec for a thermally induced stress range of 3×10^{-7} to 1.56×10^{-7} dynes per sq cm. The velocities of the screw components were found to be much less sensitive to stress. In order to compare these velocities with those measured by the etch-pit technique, dislocations ending at a free surface were also studied. Although stationary, these dislocations were heavily bowed indicating that a large surface drag force exists which must be overcome before the dislocation can glide. It would appear then that the stress for dislocation mobility measured by the etch pit technique is consistently higher than the actual stress required for dislocation motion in the bulk material. Comparison of the stress-velocity measurements on magnesium with those of Stein and Low on silicon iron crystals shows this effect is indeed significant. Observations of *Taylor locking*, forest type intersection, and an edge-screw interaction were also made.

Studies of Dislocation Density and Deformation in Single Crystals of Lead Sulfide as a Function of Stoichiometry: *Wilbur Franklin* and *J. B. Wagner, Jr.*, Yale University.

Some properties of the partially polar semiconductor compound, lead sulfide, were investigated as a function of the deviation from the stoichiometric composition. Single crystals were equilibrated at 700° and at 800°C under partial pressures of sulfur corresponding to stoichiometric, lead excess and sulfur excess compositions. The samples were quenched and the dislocation density on cleaved (100) planes was determined by an etch pit technique. A very small but reproducible variation in dislocation density with deviation from stoichiometry was observed in undoped and in silver doped crystals. Results were consistent with

theory relating the contribution of a supersaturation of atomic defects to total dislocation line length.

Observations of small parallelopiped cavities in silver-doped PbS and the dissolution kinetics of etch pits in undoped PbS are reported.

Utilizing a prismatic punch technique, the critical stress to initiate glide by pushing a prism of material through cleaved samples having (100) cleavage faces was related to deviation from stoichiometry in undoped PbS and in silved-doped PbS. Specimens having greater defect concentrations, that is, excess lead or excess sulfur, required higher stress to deform than specimens near the stoichiometric composition. The {100} glide planes, <110> glide directions and {100} cleavage planes were verified for PbS.

This work was supported by the Office of Naval Research.

Investigation of Fatigue in Metals by Means of Etch Pits at Dislocation Sites: *A. H. King,* Arthur D. Little, Inc.

Recently reported chemical etching techniques permit the observation of dislocation arrangements and movements under an applied stress in single crystals of copper. A correlation within at least 20 pct between dislocation density and observed etch pit density has been reported, and it appears possible at appropriate crystal orientations to distinguish between positive and negative edge dislocations.

These etch-pit techniques have also been found applicable to certain copper solid solution alloys, and have been used to compare dislocation movements during the early stages of fatigue in bending in copper and copper alloys of known stacking fault energy. Preliminary results will be given and the implications discussed in terms of dislocation theory.

Symposium on the Effects of Surface and Environment on Strength II

2:00 to 5:00 pm — **Shelby Room**

(Sponsored by the Committees on Corrosion Resistant Metals and Chemistry and Physics of Metals)

Chairman:

C. S. Barrett, University of Chicago.

The Interaction of Surfaces and Dislocations: *A.K. Head,* Commonwealth Scientific and Industrial Research Organization (Australia).

A theoretical analysis is made of the influence of the surface of a solid on the properties of dislocations. In particular, the effect of a surface film in impeding the escape of dislocations from the solid is considered, and the dependence of this blocking effect on the properties of the surface film. A special case of this analysis is for a dislocation in a thin film. It is shown that the properties of such a dislocation will differ significantly from those of a dislocation in a bulk solid. In addition to the thin film changing the properties of the dislocation, the presence of a dislocation may cause a bending or buckling of the thin film. The magnitude of these deformations is estimated and the possibility of their observation discussed.

The Variation of the Barrett Anomalous After Effect with Torsion: *D. B. Holt,* University of Witwatersrand

The variation with torsional strain of the magnitude of the transient in the Barrett abnormal after effect has been studied. The results provide evidence that the oxide film yields at a strain of about 1° per cm in 1 mm diam aluminium specimens, i.e. at a surface strain of about 8.7×10^{-4}, but that at much larger twists there are again stresses which can be released by acid attack on the oxide layer. The transient takes but a short time in the small twist region, but in the large twist region it takes much longer. Transverse, longitudinal, and 45° slip lines were visible on electropolished specimens, but only 45° lines were visible on anodized specimens. Electron microscopic examination showed that the 45° lines produced wide cracks in the oxide film, whereas the transverse and longitudinal lines produce detachment of the oxide film or narrow cracks in the oxide film. It appears that dislocation pile ups on the primary, transverse and longitudinal, glide systems are responsible for releaseable stresses in the region below 1° per cm and those on the 45° slip system for at least part of the releaseable stresses in the large twist region. The effect of the anodizing time on the structure and properties of the film was studied. These results are discussed in terms of dislocation theory and in relation to the literature on surface effects in the plastic deformation of metals. This work was financially supported by the South African Council for Scientific and Industrial Research.

Influence of Thin Films of Solid Nonorganic Compounds on the Strength Properties of Metals (tentative): *L. Nanis* and *J. H. Schulman,* Columbia University.

The Effect of Surface on the Mechanical Deformation of Metal Single Crystals: *Irvin R. Kramer,* The Martin Co.

When single crystals of aluminum and gold were pulled in tension while the surface layers were removed continuously by means of an electrolytic polishing bath, the extents of Stages I and II were increased and the slopes decreased as the rate of metal removed was increased. From these results it appears that the work-hardening coefficient of Stage I is determined almost entirely by the surface, while in Stages II and III, it is determined by both internal and surface obstacles. When a specimen of aluminum was deformed to within the Stage I region, it was possible to recover completely the original critical resolved shear stress and extent of Stage I by removing enough of the cross-section by electrolytic polishing; this was not possible when the specimens were deformed to within the regions of Stages II and III. These results indicate that during the deformation process *debris* is formed in regions near the surface of the crystal which impedes the passage of dislocations.

The behavior of polycrystalline specimens of aluminum, when pulled in tension while the surface layers were being removed, was very similar to that of the single crystals. Throughout the plastic deformation the slope of the stress-strain curve was lowered as the rate of removal of the metal was increased.

Studies of the tensile behavior of single crystals of aluminum and copper in nonpolar solvents containing surface-active agents show that the degree to which the extents and slopes of Stages I and II are affected varies with the concentration of the solution. The results lead to the belief that the changes in the deformation characteristics are associated with the formation of metal soaps and the rate of solution of the soaps in the solvent. Additions of a metal soap to a solution containing a surface-active agent markedly decreases the *weakening* effect of the surfactant.

The creep behavior of gold single crystals in paraffin oil was not affected by the addition of oleic acid or octanoic acid. However, the creep rate was increased markedly when the surface layers were removed continuously by means of an electrolytic polishing bath.

Role of the Surface on the Strength, Ductility and Embrittlement of Metals: *A. Berghezan,* European Research Associates.

During previous work by transmission electron microscopy on the mechanism of plastic deformation and rupture of metals, it was found that the nucleation of dislocations in the surface plays an important part in the deformation process. Further, the surface influences the rate at which dislocations generated inside the sample flow out. Hence, any transformation of the surface layer should influence drastically the mechanical properties of the metals both by influencing the nucleation of slip in the surface, and by the barrier action of the surface layer to the outflow of dislocations generated inside the crystal.

On the basis of the above observations, some measurements of the mechanical properties have been performed on some pure reactive and refractory metals possessing a clean surface or contaminated to different degrees by introduction of interstitials to different depths into the surface. Oxidation in air at different temperatures and times of oxidation provides a simple and excellent mean of hardening the surface under the oxide layer which scales off. The depth of the oxygen penetration is easily measured by microhardness indentations on a cross section.

As expected, it was found that by building up a hard brittle layer on the surface of a ductile metal, the ductile material will largely or completely lose its ductility. Vice versa, the removal of that layer restores the ductility of the metal.

MONDAY, OCTOBER 23

Although the phenomenon is quite general, three metals, one of group IV (Ti), one of group V (Cb), and the other of group VI (W) of the periodic table have been chosen as specific examples for intensive study. The influence of interstitials other than oxygen, mainly C and N are also under investigation.

This work has been sponsored by Union Carbide Corp., and this support is gratefully acknowledged here.

High Purity Iron and Its Dilute Solid Solutions

2:00 to 5:00 pm — Sky Room

(Sponsored by IMD Ferrous Metallurgy Committee)

Chairman:

Maxwell Gensamer, Columbia University.

Strain-Hardening, Recovery, and Recrystallization of Iron: *W. C. Leslie* and *J. T. Michalak*, United States Steel Corp.

Plastic Deformation and Fracture of Iron: *J. R. Low, Jr.*, General Electric Co.

Effects of Solute Elements and Some Other Variables Upon the Yield Stress and Brittle Fracture Stress of Iron: *N. P. Allen*, National Physical Laboratory (England).

TUESDAY, OCTOBER 24

Titanium in 1975

9:00 am to 12:00 m — Terrace Room

(Sponsored by IMD Titanium Committee)

Chairman:

W. W. Minkler, Titanium Metals Corp. of America

Background:

W. W. Minkler, Titanium Metals Corp. of America

Production of Primary Titanium Products:

H. H. Kellogg, Columbia University

Materials:

D. J. MacPherson, Armour Research Foundation

Continuous Casting

9:00 am to 12:00 m — Ballroom

(Sponsored by ISD Physical Chemistry of Steelmaking Committee)

Chairmen:

C. L. McCabe, Carnegie Institute of Technology, and *Robert Clark*, National Forge Co.

Attendance at sessions limited to persons registered for Metallurgical Society Fall Meeting. Registration information—Page 2.

Continuous Casting in Europe—Practice and Developments: *I. M. D. Halliday*, United Steel Companies (England).

Progress in Continuous Casting Stainless Steel Slabs at Atlas Steels: *L. Nemethy, Earl Stock, and W. B. F. McKay*, Atlas Steels Ltd. (Canada).

The paper will cover some of the metallurgical aspects of continuous casting stainless steel slabs at Atlas. The solidification characteristics of austenitic and ferritic stainless steels and certain special austenitic alloys will be discussed. The surface characteristics of the as-cast slabs will be explained next in view of the most important metallurgical and casting parameters such as casting temperature, casting speed, special alloying elements, hydrogen, type of oscillation, etc. The internal quality of the as-cast slabs will be explained in terms of chemical homogeneity, center porosity, and cleanliness.

The Weybridge Multiple Mould Process for Continuous Casting of Tonnage Steel: *J. Pearson*, British Iron and Steel Research Association, and *J. Savage*, Continuous Casting Co., Ltd., (England).

Hitherto, the casting rates of so-called billet and bloom sections produced by continuous casting have been relatively low, ranging from 5 to 25 tph per strand for sections of 2 to 10 in. square. Raising casting rates by increasing withdrawal speeds results in deterioration of the quality and a risk of failure of the casting process due to tearing or bulging of the weak ingot shell.

To obtain increased rates of casting for square sections, The Continuous Casting Co. Ltd. and The British Iron & Steel Research Assn. have successfully pioneered the development of an entirely new method of continuous casting, known as the *Weybridge* Process. This is based on a multiple mould comprising two or more square sections which are connected by a narrow channel at their corners, the mould being supplied with liquid steel from only one nozzle. This process permits a substantial increase in casting rate for any square section. The inherent problems associated with tundishes and nozzles at low rates of teeming are entirely overcome as a consequence of the increased casting rates which are now made possible.

Multiple ingots of EN 5 steel, 3 in. square, have been parted without difficulty by oxy-acetylene torch. The as-cast product, and bars 1¼ in. diam rolled from the parted sections, have been examined metallurgically. There is no significant difference between the separated ingots, and the rolled product easily met all the mechanical test requirements of the relevant B.S. 970 specification. The process yields sound commercially acceptable steel.

TUESDAY, OCTOBER 24

Symposium on the Effects of Surface and Environment on Strength III

9:00 am to 12:00 m — Shelby Room

(Sponsored by the Committees on Corrosion Resistant Metals and Chemistry and Physics of Metals)

Chairman:
J. J. Gilman, Brown University.

Some Effects of Adsorbed Polar Molecules on the Etching and Yielding Behavior of LiF Crystals: *A. R. C. Westwood*, Research Institute for Advanced Studies.

Metallographic studies have demonstrated that aqueous solutions of long chain fatty and perfluoro-acids can serve as etchants for LiF crystals revealing the points of emergence of dislocations and other surface features of interest. Polar molecules are believed to be adsorbed both at dislocations and kink sites thus affecting kink nucleation as well as kink motion.

These etchants are being used to study the cross-gliding of screw dislocations at the surface of crystals in inert and surface-active environments. On the basis of present observations, it is suggested that chemisorbed polar molecules restrict and control the motion of cross-gliding screw dislocations. Surface-active environments do not appear to assist cross-gliding by lowering the surface energy of the crystal via adsorption.

In view of this, polar molecules also might be expected to hinder the operation of the half-loop surface-sources present in as-cleaved crystals—*adsorption-locking*. An estimate of the possible magnitude of such locking has been made and its existence confirmed by studies on the yielding behavior of LiF crystals. Compression and bending experiments revealed that adsorbed polar molecules cause an increase in yield critical resolved shear stresses before inducing the relatively lower rate of work-hardening commonly observed with other materials and known as the Rebinder effect.

Possible explanations for the etching and mechanical behavior will be presented.

This work was supported in part by Watertown Arsenal Laboratories.

Effect of Surface Condition on the Mechanical Properties of Polycrystalline Ionic Solids: *R. J. Stokes*, Minneapolis-Honeywell Regulator Company.

Ionic solids of the rock salt structure belong to the *semi-brittle* class of solids. That is to say, they have the ability to deform plastically but are completely brittle in the presence of small defects. Defects can be produced in a variety of ways and only by proper control over them can the intrinsic high strength or ductility be measured. One of the most important factors controlling the initiation of flaws is the surface condition.

To illustrate this point the strength and ductility of single and polycrystalling magnesium oxide will be compared for three different surface conditions. These can be attained experimentally by a chemical polishing technique followed by controlled surface impact with tiny hard particles:

1) The *perfect* surface, i.e., chemically polished and handled so carefully that only the strongly locked grown-in dislocations remain. Both single crystals and bi-crystals are extremely strong (greater than 100,000 psi) under this condition. They fracture due to microcracks suddenly generated at the onset of plastic deformation; in single crystals at slip band intersections; in bi-crystals at the grain boundary;

2) Surfaces containing fresh dislocations only. Single crystals are relatively weak (approximately 10,000 psi) and ductile. The ductility depends upon the density of slip since the size of a microcrack formed at a slip band intersection is dictated by slip band spacing. Bi-crystals are weak and brittle. A slip band intersecting the grain boundary *immediately* nucleates a microcrack. Slip cannot be tolerated in polycrystalline magnesium oxide at room temperature; and

3) Surfaces containing fresh dislocations and microcracks. Both single and polycrystalling specimens are weak and brittle due to the propagation of surface defects. The effect of surface condition on the mechanical behavior of single and polycrystalline sodium chloride will also be discussed.

This work is supported by WADD and ONR.

The Effect of Controlled Surface Contamination on the Ductility of Ionic Crystals: *G. T. Murray*, Materials Research Corp.

It has been previously shown that the formation of a $NaClO_3$ surface layer on NaCl crystals during exposure to air, oxygen, or ozone is one of the major sources of atmospheric embrittlement in rock salt. Numerous examples of atmospheric embrittlement of MgO single crystals have also been reported, but several investigators have contradicted these findings. The present work was initiated with the object of clarifying this point and, in the event that embrittlement was found in MgO, of determining the mechanism involved.

Chemically polished MgO crystals have been exposed to varying pressures of air, O_2, N_2 argon, CO_2, and moisture, at temperatures ranging from 25° to 250°C and for periods of several days. Subsequent bend tests, conducted over a wide temperature range and on sufficient numbers of specimens to outweigh experimental scatter, have failed to show embrittlement compared to control specimens even when (as was the case for samples exposed to moisture) clearly visible surface deposits were present. These results contradict previous reports of a low-temperature atmospheric embrittlement effect in MgO.

One example of embrittlement of MgO by a surface compound has been observed. This occurred on samples which were allowed to form a surface residue during chemical polishing. The surface compound which causes the embrittlement has been identified as redeposited polycrystalline MgO.

The experiments have served to illustrate the excessive care that must be taken in preparing samples of ionic materials for mechanical testing, and in handling such samples during subsequent treatments. Lack of sufficient experimental care may be the source of the varied reports on the embrittlement of MgO by exposure to gases.

Experiments are in progress on the effect of elevated temperature gaseous exposure, the influence of gases on samples containing surface flaws, and on mechanism of residue (redeposited MgO) embrittlement.

This project was sponsored by WADD, Materials Central, Ceramic and Graphite Branch under subcontract from Armour Research Foundation.

Effect of Environment on the High Temperature Strength of Metals: *M. R. Achter*, Naval Research Laboratory.

At elevated temperature, the strength of metals in vacuum or inert gases may be either higher or lower than that measured in a reactive gas. A mechanism to explain this reversal of the atmosphere effect has been proposed based on two competing processes. Adsorption of gas facilitates crack propagation while oxidation and similar reactions harden and strengthen a metal. The controlling process is determined by temperature and stress. Current studies designed to investigate the operation of these processes are presented.

A series of experiments has been conducted to investigate the effect of gas adsorption on crack propagation in creep and fatigue. Nickel is inert toward nitrogen, yet it is appreciably weaker in this gas than in vacuum. Metallographic studies show that this reduction in strength is related to an increase in number of intergranular cracks when the test is conducted in the gas. Changes in purity of both the metal and the gas affect the results and are indicative of the mechanism of the process. The results are discussed in terms of a model in which gas adsorption at the tip of a crack reduces the work required to break intermetallic bonds.

At sufficiently high temperatures, formation of load bearing oxide layers in grain boundary cracks, it is shown, can be an important source of *oxidation strengthening*. The measured rupture strength of sintered nickel oxide layers is of the right magnitude to account for some instances of strengthening and for the occurrence of anomalous fillet fractures in nickel creep specimens.

The Effects of Elastic Strain and Environment on the Surface Free Energy of Solid Copper: *S. Floreen, D. V. Ragone*, and *E. E. Hucke*, University of Michigan.

A relationship is developed to show that the average surface free energy f^s and grain boundary free energy f^B change with uni-axial elastic strain as follows:

$$f^s = f^s{}_o + k\epsilon^2$$
$$f^B = f^B{}_o + j\epsilon^2$$

where $f^s{}_o$ and $f^B{}_o$ are the respective energies at zero strain, ϵ is the strain, and k and j are constants.

TUESDAY, OCTOBER 24

Measurements of the slopes of stress-strain curves in the elastic region for copper specimens of different sizes and grain diameters in various environments indicate the values of k and j to be of the order of 10^9 and 10^9 ergs per sq cm, respectively.

This work was partially supported under Air Force (OSR) contract No. AF 49 (638)-422.

Physical Metallurgy of Refractory Metals

9:00 am to 12:00 m — **Sky Room**

(Sponsored by IMD Refractory Metals Committee)

Chairmen:

Joseph Maltz, Department of the Navy, and *J. H. Bechtold*, Westinghouse Electric Corp.

Dislocation Substructure in Tantalum and Tungsten: *S. Weissmann* and *Y. Nakayama*, Rutgers University.

The development of a dislocation substructure in tantalum and tungsten was investigated by transmission electron microscopy. Progressive deformation led first to the formation of a cell structure, and subsequently to a substructure. The cell structure was formed by dislocation entanglements, whereby cross-slip played an important role. The substructure was formed by the interaction of entangled dislocations constituting the cell wall with entangled dislocations located within the cell.

Annealing commercial tungsten above 2500°C and subsequent quenching caused the impurity atoms to segregate on grain boundaries and dislocation networks. High-temperature annealing of commercial tungsten, with or without deformation, followed by quenching, disclosed the presence of stacking faults.

Substructure and Mechanical Properties of Refractory Metals: *Steven Allen, E. M. Passmore*, and *B. S. Lement*, Manufacturing Laboratories, Inc.

The effect of variations in substructure of W, Ta, Mo, and Cb achieved by recovery-annealing on the ductile-brittle transition temperature and other mechanical properties is being studied. Substructure is being measured quantitatively by light and electron metallography and by means of X-ray diffraction line broadening measurements. The latter are being analyzed for strain and particle size contribution to line broadening by the Warren-Averbach technique.

Flow Characteristics of Zone-Refined Molybdenum Over the Temperature Range 4.2°K to 373°K: *J. Van den Sype and R. Maddin*, University of Pennsylvania, and *A. Lawley*, Franklin Institute Laboratories.

Specpure molybdenum rods were given varying numbers of zone passes using the electron beam floating zone technique. Specimens in the form of single crystals, or coarse grained material (0.1 to 0.5 mm grain diam) were tested to fracture, in tension, at temperatures between 4.2° and 373°K. In addition, the reversible flow stress ($\sigma_{T°K} - \sigma_{373°K}$) was determined over the range 77° to 373°K.

Results show that 99.995 pct purity Mo behaves in a ductile manner in tension down to 4.2°K, with a uniform strain (polycrystals) of approximately 5 pct at this lower temperature. For a given temperature T, the term ($\sigma_{T°K} - \sigma_{373°K}$) is independent of prior strain ϵ from $\epsilon = 0$ to 0.05. The ($\sigma_T - \sigma_{373°K}$) temperature curve for material given six zone passes is significantly lower than that for one zone pass material, while the latter curve lies below that reported for arc-cast or sintered material. The data is analysed in the light of the conflicting theories concerning the temperature dependence of yield and flow stress in body centered metals.

Flow and Fracture of High Purity Tantalum-Tungsten Alloys in the Ductile-Brittle Transition Region: *D. P. Ferriss*, E. I. du Pont de Nemours and Co., and *R. M. Rose*, and *J. Wulff*, Massachusetts Institute of Technology.

Single crystals of tantalum, tungsten, and alloys thereof were grown by electron beam zone melting and tested in tension between 77° and 373°K.

Tungsten crystals were brittle at 77°K and ductile at 195°K. The addition of 20 pct Ta raised the ductile-brittle transition to room temperature. Tantalum-base alloys showed a ductile-brittle transition at 77°K with 22 pct W and at room temperature with 31 pct W. Twinning in pure tungsten and the alloys on 211 planes was observed to be a consequence of cleavage crack propagation which occurred invariably on 100 planes with much tearing and river markings.

The onset of brittle fracture in tantalum base alloys was associated not with a fundamental change in the mode of deformation, but only with the higher yield and flow stresses required by solid solution hardening. The delineation between ductile and brittle behavior in high tungsten alloys was less clearly established, although the ductile-brittle transition was raised by increasing the tantalum concentration.

In this binary system the propensity toward brittle fracture could not be correlated exclusively with the incidence of twinning, the presence of the yield point phenomenon or the steep rise of yield stress with decreasing temperature. Cleavage cracks were observed to nucleate either at the specimen surface or at sub-grain boundaries. Therefore, it is deduced that surface imperfections and sub-grain boundaries are adequate barriers to permit the coalescence of 100 dislocations to form crack nuclei.

The implication from the single crystal work is that grain boundaries in pure tungsten, as well as alloys with tantalum, constitute a barrier even stronger than the 100 dislocation alone. Hence, cracks can nucleate at higher temperatures and lower solute composition than was found for the single crystals.

Notch Sensitivity of Refractory Metals: *A. G. Imgram, F. C. Holden, H. R. Ogden*, and *R. I. Jaffee*, Battelle Memorial Institute.

The tensile and notch tensile properties of four refractory metals (molybdenum, tungsten, columbium, and tantalum) and one alloy (Mo-0.5Ti) were investigated. All the materials were evaluated in bar form, and the molybdenum and Mo-0.5Ti also were studied in sheet form. The notch sensitivity of each material was evaluated on the basis of several criteria, including the notch-unnotch strength ratio, the ductility transition, and the fracture transitions.

Effects of Dispersions on the Recrystallization and Ductile-Brittle Transition Behavior of Tungsten: *R. I. Jaffee, B. C. Allen*, and *D. J. Maykuth*, Battelle Memorial Institute.

Sintered tungsten-base alloys with up to 10 volume pct of fine oxide may be fabricated to strip by conventional hot-working procedures. ThO_2, ZrO_2, Al_2O_3, and UO_2 are stable, while SiO_2 and MgO are volatilized during vacuum sintering. TiN, ZrN, HfN, TaN, HfC, TaC, and Ta_2C are not stable, with nitrogen, carbon, and titanium being evaporated in vacuum sintering, leaving the residual metallic elements. Residual tantalum and hafnium are detrimental to fabricability, but zirconium is not. Primary recrystallization temperatures are raised as much as 200°C by ThO_2 + Na_2O dispersion, ZrO_2, and residual Zr or Ta. Secondary recrystallization was retarded up to over 2700°C with the ThO_2 + Na_2O dispersion. The ductile-brittle transition in bending could be lowered as much as 90° by dispersions, with ThO_2 + Na_2O most effective.

Yield and Fracture in Polycrystalline Tantalum: *M. A. Adams*, Materials Research Corp.

Tensile tests have been made on specimens of commercial tantalum of different grain-sizes, and the variation of lower yield stress σ_y with grain-size 2d has been used to assess the effect of temperature (23°, −78° and −196°C) and strain-rate (1.09×10^{-2} and 9.96×10^{-2} per sec at each temperature) on the parameters σ_i and k_y in a Petch type equation, $\sigma_y = \sigma_i + k_y \cdot d^{-1/2}$. The results indicate that the sensitivity of the yield strength to temperature and strain-rate arises almost entirely from the effects that these variables produce on σ_i, which is in agreement with observations for other body-centered-cubic transition metals. The high resistance of tantalum to brittleness has been confirmed, all of the specimens showing pronounced necking and predominantly fibrous fractures; however, on the fracture surfaces of the coarser grained specimens tested at −196°C (both strain-rates) some cleavage facets were observed. This observation of cleavage can be adequately explained by Cottrell's transition equation $(\sigma_i\, d^{-1/2} + k_y)\ k_y = \beta \cdot \mu \cdot \gamma \cdot$ when account is made of the effect of deformation on the parameters in the equation, and a value of

TUESDAY, OCTOBER 24

the effective surface energy for fracture γ is obtained ($1.35 \times 10^4 \geq \gamma > 1.16 \times 10^4$ erg cm^{-2}) which is in the same range as those values which have been previously measured for columbium, molybdenum and mild steel. The greater ductility of tantalum relative to these materials is attributed to a low value of k_y and a high value of the shear modulus, μ. Little or no twinning was observed in the tests, probably because the material was not of a very high purity.

Titanium in 1975

2:00 to 5:00 pm — **Terrace Room**

(Sponsored by IMD Titanium Committee)

Chairman:

W. W. Minkler, Titanium Metals Corp. of America

Processes:

P. J. Rieppel, Battelle Memorial Institute

Aerospace Applications:

Dana Moran, Aerospace Industries Association of America

Terrestrial Applications:

W. E. Lusby, Jr., E. I. du Pont de Nemours & Co., Inc.

Working of Cast Structures

2:00 to 5:00 pm — **Ballroom**

(Sponsored by ISD Mechanical Working Committee)

Chairmen:

T. E. Dancy, Jones & Laughlin Steel Corp., and *C. F. Schrader,* Inland Steel Co.

Evaluation of Concast Steel for Hot Piercing Operations: *H. B. Emerick,* Jones & Laughlin Steel Corp.

This paper describes some of the technical and economic incentives for continuous casting of steel, and presents results of plant investigations designed to assess the suitability of Concast semi-finished section of eletric furnace steel for the manufacture of seamless pipe by the Mannesmann hot piercing process. Among the characteristics evaluated are chemical homogeneity, surface quality, internal structure, piercing quality, and mechanical properties of the resultant tubular product.

Refinements in casting techniques are indicated to be necessary, especially on the higher carbon grades of steel, in order to minimize the occurrence of stress cracks and entrained slag which lead to excessive rejections in severe hot tube piercing operations.

Effects of Forging and Rolling on Cast Steels: *Cyril Wells,* Carnegie Institute of Technology.

The paper will be based on extensive work done on SAE steels from 0.30 to 0.45 pct C and 1.5 to 4.5 pct total alloy content. It will discuss the relation between ingot size and reduction of area to physical properties, and include the effect of segregation and non-metallic inclusions. Although the work was based mainly on ingots, the paper will be written from the point of view of its relevance to the working on continuously cast material.

The Role of High Strain Rates and Temperature on the Deformation and Fracture Characteristics of Iron-Carbon Alloys: *Walter Lawson, T. B. King,* and *N. J. Grant,* Massachusetts Institute of Technology.

This will cover a study of fracture ductility of cast, iron-carbon alloys (0.01-1.35 wt pct C) in relation to stress, strain rate and temperature. Stress was varied from 1000 to 1800 psi, strain rate from 0.001 to 100 pct per sec, and temperature from 1600° to 2200°F. These conditions approach those found in hot-working processes.

Vacuum Techniques in Metallurgy

2:00 to 5:00 pm — **Shelby Room**

(Sponsored by EMD High Temperature Metals Committee)

Chairmen:

W. W. Gullett, Chicago Development Corp., and *W. H. Dresher,* Union Carbide Nuclear Co.

Arc vs. Electron Beam Melting in High-Vacuum Environments: *R. P. Morgan* and *T. E. Butler,* Union Carbide Metals Co.

In recent years, the electron beam-melting technique has attained a dominant position in the field of refractory metal consolidation where an end-product of exceptional chemical purity is desired. This position reflects the ability to operate the melting process at ambient pressures of 10^{-4} mm or less, thereby, taking advantage of a substantial improvement in the kinetics of removal of non-metallic and metallic impurities.

The present paper reviews some of the problems which were encountered in an attempt to operate the consumable-electrode arc under equivalent pressure conditions in order to achieve similar purification benefits. Factors of arc instability, control of material processing rates, and chemical purity of the ingot product are discussed, with particular reference to the behavior of columbium, tantalum, and tungsten. It is concluded that, with certain reservations, equivalent product purity can be obtained by either melting technique.

Production Applications of the Electron Beam Heating Source to the Refractory Metals: *E. F. Baroch* and *W. A. Aschoff,* Wah Chang Corp.

The most familiar application of electron beam to processing refractory metals is in melting. Most of the furnaces in use today are the Temescal type in which a close-proximity, work-accelerated gun is employed. These units are especially useful for the refractory metals as their melting points and relatively low vapor pressures allow vacuum purification. Generally, any material with a vapor pressure ten times that of the material being melted can be removed by electron beam melting in a high-vacuum environment. In addition to their relative vapor pressures, other factors entering into the purification are the equilibrium concentration in the molten state and physical bonding forces. Typical examples are the ease of removal of carbon and oxygen as carbon monoxide from columbium and the ability to remove practically all metallic

The Metallurgical Society of AIME

FALL DINNER

Tuesday, October 24—Terrace Room

6:30 pm Social Hour for Holders of Dinner Tickets (Sponsored by Detroit Section, AIME)
7:00 pm Fall Dinner
Toastmaster: J. Harry Jackson, Chairman, Institute of Metals Division
Welcome: E. D. Marande, Chairman, Detroit Section, AIME
Guest Speaker: Dr. Chauncey D. Leake, Chairman, Board of Directors, American Association for the Advancement of Science
Topic: Standards of Measurement and Nursery Rhymes

Tickets ($6.50) must be purchased before 2 pm on day of dinner.

TUESDAY, OCTOBER 24

impurities from tungsten. This ability to purify by high vacuum severely limits the ability to produce electron beam-melted alloys containing high vapor pressure constituents. In addition to the ability to purify by high vacuum, the electron beam process offers further advantages such as close melt rate control since the operator has the ability to proportion the energy almost at will between the melt stock and the molten pool. Through clever design the electron beam melting furnaces operate almost continuously through the use of load locks, ingot cooling locks, and gun locks which allow insertion of melt stock, removal of finished ingots, and maintenance on the guns without disturbing the integrity of the main vacuum chamber.

Developments now in process indicate the future trends of electron beam furnace design. First will be toward larger units, such as the 1.5 Mw unit being built by Temescal Metallurgical Corp., which should be capable of melting ingots up to 20 in. diam and which should drastically reduce costs of electron beam melting. The second design trend is in the heat source itself. Undoubtedly, we will see remote guns which are self-accelerated to give longer gun life, better focusing, hotter, deeper pools, and more general versatility.

The second widespread industrial application of electron beam to refractory metals is in welding. The current practice involves three distinctly different types of operation. The first we may classify as the fine and clean as exemplified by welding very narrow beads in thin, clean, sheet material. The second—almost a direct opposite—is the rough and dirty type of welding encountered in fabricating electrodes for electron beam or arc melting. The third aplication involves sidewall fusion conditioning vacuum-melted ingots where the conditions encountered by the electron beam welder are intermediate compared to the first two applications.

Finally, the electron beam heating source is being applied to the refractory metals for sintering, annealing both batch and continuous, and for high-temperature testing.

The Use of Electron Beams in Vacuum Metallurgical Processes: *J. C. Simons, Jr.*, and *P. L. Raymond*, National Research Corp.

Electron beam technology provides advantageous heat sources for metal evaporation, encapsulation, and melting under vacuum. These electron bombardment processes are particularly applicable to the heating of refractory metals. The basic technology of electron beams is reviewed. A vacuum facility of improved design which employs three stages of differential pumping to permit pressure gradients on the order of 200:1 is described. Results of operations with electron beam heating of metals are presented for such work as: vacuum evaporating refractory metals to achieve coatings on metal surfaces; vacuum encapsulating solid materials in particulate form to alter their effective properties; and vacuum melting of refractory metals for laboratory and commercial programs.

Purification Techniques for Arc-Melting Tantalum and Tantalum Base Alloys: *M. L. Torti* and *C. A. Hornell*, National Research Corp.

Chemical analyses before and after melting a series of ingots show a slow melting rate to be an important condition for maximum purification.

A series of ingots cast using a retractable mold-base shows marked improvement in homogeneity and purification compared with a series using a conventional fixed mold-base.

The melting behavior and ingot purification obtained for various alloy compositions is reported.

Furnace Reactions With Gases Which Affect the Quality of Vacuum Induction-Melted Uranium: *R. W. Endebrock* and *E. L. Foster*, Battelle Memorial Institute.

The soundness and purity of uranium fuel slugs is dependent to a considerable extent upon process variations in melting and casting. Interaction of air and water with both uranium and the graphite container components during vacuum-induction arc melting is of particular concern because oxygen, nitrogen, and especially, CO play an important part in final metal purity. While some of these gases can be instrumental in the formation of protective drosses, high CO partial pressures over the melt can result in higher carbon-oxygen levels in uranium than would result from simple metal-gas reactions. Water is particularly troublesome in that it can react with carbon components and, thus provide large quantities of CO.

The Production of Pure Molybdenum and Its Alloys by Floating Zone Melting: *J. A. Belk*, The War Office (England).

High-purity molybdenum has been produced by the electron-bombardment, floating-zone melting method. Analysis has shown that the gas content is considerably reduced and that the reduction of carbon is mainly due to zone refining. The mechanical properties of the purified material in single crystal and polycrystalline form are given. Methods of making alloys of the refractory metals are described and their results discussed.

Physical Metallurgy of Refractory Metals

2:00 to 5:00 pm — **Sky Room**

(Sponsored by IMD Refractory Metals Committee)

Chairmen:

E. I. Salkovitz, Department of the Navy, and *H. R. Ogden*, Battelle Memorial Institute.

The Effect of Purity on the Strength of High Purity Tungsten at Elevated Temperatures: *J. L. Orehotsky*, General Telephone and Electronics Laboratories, Inc.

Since purifying tungsten by molten zone traversal lowers the transition temperature significantly and produces a material that is fairly ductile at room temperature, and evaluation was made of the effect of this amount of purification on the high-temperature strength properties of tungsten. Undoped tungsten rods, before and after zone purification, were drawn to wire and these wires, along with doped tungsten wire, were used for the evaluation. The wires were all recrystallized to the same equiaxed grain size and then stress rupture and tensile tested at temperatures between 800° and 1700°C. At the higher temperatures of testing, the strength properties were found to vary with the purity of the material. The purest wire had the lowest strength characteristics.

The high temperature strength characteristics of the as drawn cold-worked wires were evaluated also. The strength properties of the cold worked wires at elevated temperatures were excellent provided recrystallization did not occur during the testing procedure. Since *in situ* recrystallization occurred when testing the undoped and purified materials at temperatures above 1500° and 1100°C respectively, the cold worked doped wires had the best strength properties above 1500°C.

The Effects of Interstitials on the Properties of Cb-Rich Alloys in the Cb-W-Zr System: *J. W. Clark*, General Electric Co.

This paper describes the results of an investigation undertaken to more firmly establish the effects of individual interstitial elements on the mechanical properties of wrought alloys in the Cb-rich portion of the Cb-W-Zr system, and, in particular, to study the effects of simultaneous variations of the Zr and C levels. Emphasis has been placed upon the determination of creep-rupture behavior in the range 1100° to 1200°C, ductility at room temperature and upon the correlation of these properties with microstructural characteristics.

The results demonstrate that, at Zr levels of 0.5 to 2.0 pct, the optimum combination of creep resistance and low-temperature ductility is obtained at Zr:C atomic ratios near unity. At these Zr and C concentrations, a fine, uniformly distributed dispersion is observed. Compared to the low-interstitial Cb-W-Zr ternary, secondary creep rates are reduced by two to three orders of magnitude by this fine dispersion. In an alloy designated AS-30, the 100 hr rupture at 1093°C (2000°F) is increased from approximately 25,000 to greater than 40,000 psi and total elongations of greater than 10 pct are retained in room temp. tensile tests. Although creep-rupture strengths are further increased at Zr:C ratios somewhat less than 1.0, the low-temperature ductility is impaired by excess carbon and can be related to the appearance of large, banded carbides. Conversely, significant excess of Zr with respect to C results in increased ductility at low temperatures but has an adverse effect on elevated-temperature creep.

Limited data also indicate that relatively small nitrogen additions, of the order of 600 to 700 ppm, markedly reduce room temperature ductility with only moderate strengthening at high temperatures whereas large (up to 3000 ppm) additions of oxygen, added to the melt as Cb_2O_5, affect neither ductility nor creep of the wrought alloys to any significant extent.

TUESDAY, OCTOBER 24

Strengthening of Mo-Ti-Zr Alloys With Carbon and Boron: *Marion Semchyshen* and *R. Q. Barr*, Climax Molybdenum Co.

It has long been recognized that, in the case of certain dilute molybdenum-base alloys, strength properties were developed that could not be attributed solely to solid solution effects. Work with a variety of alloys containing titanium and/or zirconium brought out the fact that the properties were sensitive to the residual carbon content present. Strength properties of wrought, arc-cast alloys containing these elements have been found to vary significantly depending on the ratio of carbon-to-titanium, or carbon-to-zirconium present. In both systems, the optimum carbon content has been shown to be approximately 1/10 that of the alloying element. A similar relationship has been observed for alloys in which carbon is replaced by boron. In the case of the latter group of alloys, however, the general level of strength properties were lower than for carbon-containing materials.

Tests on a series of molybdenum-base alloys containing columbium and carbon indicate that strength properties are not sensitive to the C:Cb ratio.

The strength dependence of molybdenum-base alloys containing titanium and/or zirconium with carbon has been attributed to the locking of dislocation movement by the critical dispersion of the carbide phases.

Effect of Heat Treatment and Precipitation Hardening on Strength Properties of Mo-Base Alloys: *W. H. Chang* and *L. P. Jahnke*, General Electric Co.

A previous investigation has established conclusively the precipitation hardening process in the Mo-TZC (Mo-1Ti-0.1Zr-0.14C) alloy. The hardening process was shown to be associated with the precipitation of TiC accompanied by simultaneous dissolution of pre-existing Mo_2C upon aging.

In this paper, the effect of heat treatment on the tensile and rupture strength of Mo-TZC in both worked and non-worked conditions, is discussed. The worked condition is shown to be greatly strengthened by prior high-temperature annealing or annealing and aging. In the non-worked condition, low-temperature strength is increased by aging, whereas high-temperature strength is enhanced by annealing alone to the extent that it is equal to the strength of the worked condition. The temperature and strain-rate dependencies of Mo-TZC in various annealed or annealed and aged conditions have been investigated. In contrast to the aged condition the annealed conditions are found to exhibit reversed temperature dependency and negative strain-rate sensitivity.

The experimental observations have generally been correlated to the state of carbide dispersion as affected by heat treatment. The interplay between strain hardening and precipitation hardening is discussed.

The Effect of Yttrium and Selected Rare Earth Additions on the Removal of Interstitials from Tungsten and Molybdenum: *D. T. Klodt* and *J. F. Nachman*, University of Denver.

Continuously cast, small-diameter molybdenum and tungsten alloy ingots were prepared by melting compacted powder charges of the base metals and yttrium or selected rare-earth metals in a non-consumable electrode arc furnace. Grain refinement, reductions in as-cast hardness, and increased ingot soundness were noted in most of the molybdenum alloys but were not observed in the tungsten alloys. The ingots were machined and swaged into rods from which torsion specimens were prepared. Recrystallization studies and torsion tests were conducted. The efficiency of the removal of interstitial elements by the yttrium or rare-earth additions was evaluated by torsion ductility and ductile-to-brittle transition tests. Data are presented for a number of molybdenum alloys and the current research in progress on tungsten alloys is discussed.

The Physical and Mechanical Properties of a Chromium Base Alloy: *R. E. Cairns, Jr.*, New England Materials Laboratory, and *N. J. Grant*, Massachusetts Institute of Technology.

Some of the more important physical and mechanical properties of a chromium-base alloy containing 1 pct Cb and 0.25 pct Y were studied in detail. The brittle-to-ductile transition temperature was investigated with the alloy in the recrystallized condition and after various amounts of prestrain. The dynamic modulus of elasticity was determined by measuring the fundamental frequency of a bar in transverse vibration over the temperature range 70° to 2000°F. The high-temperature strength of the alloy was measured as a function of stress and time at 1500°, 1800°, and 2000°F in the recrystallized condition and 1500°, and 1800°F in the prestrained condition. The oxidation resistance of the alloy was studied by both the weight gain per unit area and the depth of oxygen penetration techniques.

Electron Metallographic Techniques for Studying Tungsten Wires and Powders: *C. F. Tufts*, General Telephone and Electronics Laboratories, Inc.

Any precise microscopical study of tungsten wires and powders should include examination by electron metallography. For these materials, adequate observation of substructure requires a resolving power of better than 100 Å thus making electron optical methods obligatory. In addition, replication methods of electron metallography make possible the precise determination of size and habit for aggregates and single particles contained in powder specimens.

Recent advances in technique using the carbon replica method for the electron metallography of tungsten wires and powders are discussed and illustrated. Experimental details are given for specimen preparation using platinum pre-shadowing in conjunction with Parlodion-carbon and direct-carbon replication. Illustrations include examples of substructure in cold-worked, annealed, and recrystallized wires; geometry and elements of surface geography for aggregates and single particles in powder specimens where maximum grain size can reach the order of 40 microns; and the substructure on surfaces produced by fracturing wires.

WEDNESDAY, OCTOBER 25

Nuclear Fuel Reprocessing I: Pyroprocessing

9:00 am to 12:00 m — **Terrace Room**

(Sponsored by EMD Nuclear Fuel Reprocessing Committee)

Chairmen:

W. D. Wilkinson, Argonne National Laboratory, and *R. E. Grace*, Purdue University.

WEDNESDAY, OCTOBER 25

Phase Relations in Plutonium Halide Binary Fused Salt Systems: *J. A. Leary* and *K. W. R. Johnson*, Los Alamos Scientific Laboratory.

Recent application of fused salt technology to plutonium processing has stimulated a corresponding interest in phase relationships of these systems. Binary phase diagrams for the systems $PuCl_3$-LiCl, -NaCl, -KCl, -RbCl, -CsCl, -$MgCl_2$, -$CaCl_2$, -$SrCl_2$, and -$BaCl_2$ have been determined over the complete composition range. As a general rule, the larger the ionic radius of the alkali and alkaline earth cation, the greater the tendency to form compounds with $PuCl_3$. Thus systems containing LiCl, NaCl, $MgCl_2$, or $CaCl_2$ are represented by a simple phase diagram with no compound formation. All other systems contain at least one compound.

The $PuCl_3$-KCl system exhibits two compounds, K_2PuCl_5 melting incongruently at 610°C, and K_3PuCl_6 melting congruently at 685°C. The $PuCl_3$-RbCl system reportedly contains three compounds; $RbPu_2Cl_7$ and Rb_2PuCl_5 melt incongruently at 584° and 560°C, respectively. Rb_3PuCl_6 melts congruently at 774°C. Two compounds were detected in the $PuCl_3$-CsCl system; $CsPu_2Cl_7$ and Cs_2PuCl_5 melt congruently at 616° and 625°C, respectively.

Only compounds of the type M_2PuCl_5 were found in the alkaline earth chloride systems. Sr_2PuCl_5 and Ba_2PuCl_5 melt incongruently at 628° and 759°C, respectively.

The PuF_3-NaF system contains the compound $NaPuF_4$, which indicates increasing tendency for compound formation as the size of the halide ion decreases.

Processing of Uranium Dioxide Fuel by the Salt Cycle Process: *K. M. Harmon*, General Electric Co.

Progress is reported in the development of the Salt Cycle process for uranium dioxide fuels, in which a key feature is the production of fuel-grade UO_2 by cathodic reduction of uranyl chloride dissolved in molten chloride salts.

Results are presented on the closure of the fuel cycle via this process and on studies of the electrolytic reduction of uranyl ion to UO_2, on methods for incorporating plutonium dioxide or thorium dioxide in the deposited UO_2, and on methods for production of large crystals of UO_2 and self-supporting UO_2 ceramic compacts by this process.

Work performed under Contract No. AT(45-1) 1350 for the US Atomic Energy Commission.

Fused Salt Oxidation-Reduction Process for Plutonium Fuel Recycle: *J. G. Reavis*, Los Alamos Scientific Laboratory.

A chloride conversion cycle has been evaluated as a pyrometallurgical process for plutonium-rich metallic fuel. The impure fuel is reacted with zinc chloride in sodium chloride solution in the 500° to 860°C temperature range. Plutonium and electropositive fission products are converted to the respective chlorides, which are soluble in the molten salt phase. Less electropositive elements such as molybdenum and ruthenium dissolve in the resulting liquid zinc phase. Volatile elements and volatile chlorides such as zirconium chloride, separate into the gas phase.

After separating the liquid zinc phase from the salt phase by filtration, plutonium chloride in the salt phase is reduced to recover the plutonium. The ternary plutonium-cerium-cobalt liquid metal fuel can be regenerated directly by reduction with cerium-cobalt alloy.

Recent Developments in the ORNL Volatility Process: *C. E. Guthrie*, Oak Ridge National Laboratory.

In the ORNL Volatility process, uranium-zirconium alloy fuel elements are dissolved with HF in a molten fluoride salt; the molten salt containing UF_4 is then transferred to another vessel from which the uranium is recovered as volatile UF_6 by contacting with fluorine. A pilot-plant demonstration of the process is currently underway at Oak Ridge National Laboratory. *L*-nickel is being used for the fluorinator and INOR-8 for the dissolver. The salt has evolved from a two-component NaF-ZrF_4 system to a more complex mixture in order to lower the operating temperature and thereby decrease corrosion in the system. Alternate materials of construction are being evaluated in an effort to find improved materials and a material which will stand both dissolution and fluorination conditions so that a single vessel can be used. The process is also capable of recovering uranium from BeO and ZrO_2 fuel types which may be difficult to process by aqueous methods.

Liquid Metal Processing of Fuel from the Second Experimental Breeder Reactor: *R. D. Pierce* and *Leslie Burris, Jr.*, Argonne National Laboratory.

The utilization of liquid metal solvents for processing reactor fuels is under study at Argonne National Laboratory. The main objectives of this development are reduction in fuel cycle costs and attainment of processes more suitable to certain fuels than existing processes. The present work is aimed at meeting needs encountered in the EBR-III fuel cycle. The eventual incorporation of these processes in the EBR-II fuel cycle facility will enable an evaluation of their technical and economic feasibility.

A simple melting process, melt refining, provides for quick recovery and recycle of the bulk of the discharged EBR-II fuel. A process employing zinc-magnesium solvent combinations is under development for recovery of uranium remaining in the melt refining crucible in the form of oxide and unpoured metal. A similar process has been developed for the separation of plutonium bred in the uranium blanket of the reactor.

The processes, in general, utilize favorable solubility and thermodynamic relationships to effect product recovery and the desired separations from impurities. Fused salts in combination with liquid metals are employed to enhance heterogeneous reaction rates, for example, the reduction of uranium oxides by magnesium in the zinc solution. A considerable amount of fundamental data has been obtained in support of engineering developments.

Small-scale demonstrations of the proposed processes have been made. The experience with liquid metal-fused salt systems in effecting chemical reactions, precipitations, phase separations, distillation, and product recovery is reviewed. Although the present applications are specific, the processes are of general utility and applications to other fuels are suggested.

The Fluidized Bed in Radioactive Waste Disposal: *J. I. Stevens*, Phillips Petroleum Co.

The international emphasis in research and development work on disposal of radioactive wastes is increasingly on reduction-to-solids techniques. Several processes are being investigated extensively by USAEC contractors. Considerable work both in the laboratory and pilot plant has been done on the fluidized bed, and a 60-gph demonstrational waste calcining facility utilizing this technique is undergoing cold testing at the Idaho Chemical Processing plant.

The results of pilot-plant work on the calcining of wastes from aluminum-type fuels over the temperature ranges of 250° to 550°C with fluidizing velocities from 0.8 to 1.0 fps are presented. Other independent variables investigated in the pilot plant have included feed composition, designs and operating conditions for pneumatic-atomizing feed nozzles, and return of fine particulates to the bed. The effects of these variables on the product bulk density and intra-particle porosity, the generation of small particulates, the mass median particle diameter of the product, the formation of different crystalline forms, the nozzle erosion rates, and the residual nitrate content of the product are reported. The results of a short test of a different nozzle configuration in a calciner heated by high temperature fluidizing gas are presented. The results of laboratory work on crystal phase transformation of alumina are discussed. The process flowsheet and design consideration of the demonstrational waste calcining facility are presented and the results of the first runs with simulated aluminum waste are described.

Work done under Contract AT(10-1)-205 to the US Atomic Energy Commission.

Work Hardening and Strengthening Mechanisms

9:00 am to 12:00 m — **Ballroom**

(Based on Submission of Research Abstracts)

Chairmen:

C. Elbaum, Brown University, and *R. W. Guard*, General Electric Co.

Strengthening Mechanisms and Alloying Theory for Body Centered Cubic Metals: *D. P. Gregory* and *G. H. Rowe*, United Aircraft Corp.

The strength of a metal or alloy is governed by the ease or difficulty with which large numbers of dislocations are able to move through the lattice by glide or climb. At temperatures above about 0.15 of the absolute melting temperature, the resistance to dislocation motion in very pure bcc single crystals is not large.

WEDNESDAY, OCTOBER 25

The pure metals can be strengthened by cold working, grain size refinement, or by introducing point defects by quenching or irradiation. Loss of strength, however, occurs as these metals are heated to 0.35 to 0.4 of the absolute melting point. For this reason pure metals are alloyed to introduce dislocation barriers which have greater temperature stability.

There are basically three mechanisms for strengthening pure metals by alloying. The first involves pinning the dislocations so that they either cannot move at all, or if they do move a frictional drag results from the movement. Cottrell atmospheres, precipitates in dislocations, and Suzuki segregation fall in this first classification. The second mechanism places barriers in the path of moving dislocations so that they must bend, climb, cross slip, etc., to pass the barriers. Mechanisms such as the Mott-Nabarro effect, cluster hardening, Fisher hardening, dispersion hardening, grain and sub-grain boundary strengthening, and precipitate stabilized networks of forest dislocations fall in this second classification. The third mechanism concerns alloying to slow dislocation climb rates. Mechanisms of modifying the energies to form and move lattice vacancies, and pinning the jogs in dislocations which absorb vacancies fall in this latter class.

Recently strengthening mechanisms have been studied in numerous alloys directly by transmission electron microscopy. Many of the strengthening mechanisms listed above have been identified in these studies. The authors propose to discuss these current concepts of strengthening mechanisms and to apply the principles to alloy design of the Va and VIa bcc metals. It will be shown that optimum low temperature tensile and elevated temperature creep properties are obtained when these metals are alloyed to utilize two or all three of the general mechanisms described above. The temperature stability and temperature potential of the various mechanisms will be discussed.

Transmission electron microscope studies of strengthening mechanisms in columbium containing various levels of interstitial impurities and columbium containing 1 pct Zr will be presented in the light of the theory described above.

This work was performed in the CANEL Materials Laboratory of Pratt and Whitney Aircraft Division, United Aircraft Corporation, under contract to the United States Atomic Energy Commission.

Work Hardening Mechanisms in Polycrystalline Columbium: *D. P. Gregory* and *G. H. Rowe*, United Aircraft Corp.

The high melting point of the bcc refractory metals makes them very attractive for high temperature structural applications. When pure, however, these metals exhibit relatively low rates of work hardening. From the structures standpoint this is undesirable because a lack of work hardening results in structural instability. A study of dislocation mechanisms which lead to work hardening in bcc metals has been initiated to explain this low hardening rate.

Dislocation density increases rapidly with plastic strain in metal crystals. Stein and Low have shown that macrascopic yielding in bcc silicon iron single crystals occurs when a large number of dislocations achieve a critical velocity. If this is generally true for the bcc system, work hardening results from:

1) decrease in the number of mobile dislocations with increasing strain,
2) decrease in the velocity of mobile dislocations with increasing strain, and
3) decrease in both the number and velocity of mobile dislocations with increasing strain.

Specific dislocation mechanisms of work hardening proposed in the literature fall into three general classifications:

1) Exhaustion of dislocation sources;
2) Interaction between strain fields of parallel dislocations:
   a) Taylor theory of work hardening
   b) back stresses from dislocation pile ups
   c) strain energy of a slightly distorted cross grid of screw dislocations; and
3) Interaction between mobile and forest dislocations during intersection:
   a) energy to form jogs and kinks
   b) non-conservative motion of jogs in screw dislocations
   c) attractive junctions lead to recombinations between mobile and forest dislocations.

For any mechanism the stress would have to increase with increasing strain either to generate new dislocations, to release immobilized dislocations from barriers which have arrested their motion, or to push mobile dislocations through an increasing density of forest dislocations.

Results of activation energy, activation volume, surface slip marking, and transmission electron microscope studies with pure polycrystalline columbium will be presented. The surface marking and transmission electron microscope studies show where the dislocations in individual crystals are located (as free dislocations, in sub-boundaries, in Frank nets, in tangled networks, or associated with precipitates) and how the dislocation density is distributed between these various locations as a function of strain, temperature, crystal orientation, and strain rate. The results are discussed in the light of the work hardening theories listed above.

This work was performed in the CANEL Materials Laboratory of Pratt and Whitney Aircraft Division, United Aircraft Corporation, under Air Force sponsorship.

Latent Hardening Experiments and Work-Hardening Theories: *U. F. Kocks*, Harvard University.

The influence of the operation of one glide system upon the subsequent operation of another (on an *intersecting* plane) is investigated by two methods: uniaxial compression of a cube in two directions; and combined torsion and compression (in varying ratios) of thin-walled prismatic (square or hexagonal) single crystals. Emphasis is placed on the exact relationship of the two systems (conjugate, cross-slip, etc.) and on the implication of these results on existing work-hardening theories.

Strain Hardening of Zinc Crystals in Alternating Tension and Compression: *T. H. Alden*, General Electric Co.

Zinc single crystals of several orientations were tested in alternating tension and compression to a plastic shear strain amplitude of 1 pct, to determine the nature of the strain hardening under these conditions. Curves were plotted of the stress amplitude vs the accumulated plastic strain.

Crystals with a basal plane normal—specimen axis angle (ϕ) of 45° were tested at room temperature with strain rates from 3.5 to 35 pct per min. Slow linear hardening to shear strains of more than 3000 pct followed rapid initial hardening. With increasing strain rate, the slope of the curve at a given strain was increased, the slope of the linear portion varying from about 5 to 15 psi at the strain rate extremes.

More detailed study was carried out at −52°C, a temperature low enough to prevent appreciable static recovery. For ϕ between 25° and 65° the shear stress amplitude, shear strain curves were similar, showing approximately linear hardening with a slope of 800 to 1000 psi for the entire curve. The slope increased only slightly with strain rate. Despite a grip to gauge area ratio of about 6 to 1, tests were normally terminated by grip failures. Under the most favorable gripping conditions, crystals hardened from an initial yield stress of 50 psi to more than 3500 psi over a shear strain of about 350 pct. In these extended tests, it appeared that the hardening slope was decreasing near the termination of the curve.

The behavior at −52°C differs from that of copper, tested under similar conditions, by the relatively high stresses to which the hardening remains linear. Continued linear hardening reflects the difficulty of dynamic recovery in zinc by the mechanism of cross slip. An approximate non-hardening plastic strain condition, suitable for fatigue fracture, cannot be established. Instead, failure occurs by an alternate mode, in this case cleavage. These conclusions are in agreement with the observed difficulty of fatigue fracture in zinc at low temperature.

Fine Particle Effect in Dispersion-Strengthening: *G. S. Ansell*, Rensselaer Polytechnic Institute.

Most of the theoretical treatments of the effect of a dispersed second phase upon the yield strength of dispersion strengthened alloys have predicted a relationship of mean free path between dispersed particles to yield strength. Recently Meiklejohn and Skoda, investigating alloys consisting of very fine spherical particles of iron dispersed in a mercury matrix have shown a strengthening effect which appears incompatible with current theories for yielding in dispersion strengthened alloys. In particular, for a series of these alloys which contain particles whose diameters range from 50 to 770A they found that the offset yield stress is only a function of the volume fraction of the second phase and bears no relationship to the inter-particulate spacing.

That this apparent anomaly between the experimental data and theory may be resolved is shown in the following manner. Ansell and Lenel proposed a dislocation model for dispersion strengthening in which yielding occurs in these alloys when the second phase particles fail due to shear stress associated with dislocations piled up around or against the dispersed phase particles. On the basis of this model they showed that for alloys containing particles whose radius of curvature was large enough to consider the piled up arrays of dislocations as straight, the yield strength should vary as the reciprocal square root of the dispersed particle spacing.

In this paper, the yielding behavior of alloys containing particles where the radius of curvature is so small, that the array of dislocations can no longer be considered straight, is treated.

WEDNESDAY, OCTOBER 25

Calculations based upon this *fine particle dispersion* model predict that the yielding behavior of these alloys is independent of interparticulate spacing and is only a function of the volume fraction of the dispersed phase. Consideration of this effect not only appears to explain the experimental data obtained by Meiklejohn and Skoda, but also appears to account for the change of yielding behavior observed during aging of precipitation strengthened alloys.

Electron Microscope Observations on Deformed Copper Single Crystals: *Z. S. Basinski* and *D. B. Dove*, National Research Council (Canada).

Copper single crystals have been deformed in a tensile testing machine prior to examination by electron microscopy. Specimens for transmission microscopy were prepared from the deformed crystals by electrolytic machining and electropolishing techniques.

Dislocation *tangles* characteristic of room temperature deformation were observed after deformation at temperatures down to 4.2°K, providing evidence that the tangles form by a geometrical rather than diffusional mechanism. Dislocations in crystals deformed in single glide show a strong tendency to form layers of tangles parallel to the main glide plane, and to a lesser exent parallel to other (III) planes. Preliminary attempts at dislocation configuration analysis by burgers vector determination, indicate that the accumulating dislocations are *not* those corresponding to the main glide system. Observations on replicas and on specimens thinned from one side are also presented.

Work Hardening and Dislocation Arrangements: *J. C. M. Li*, United States Steel Corp.

Contributions to work hardening from the long-range stress field of dislocations of one slip system or of the tangles when dislocations of two slip systems interact, as well as contributions from the shear and the motion of the cell wall will be discussed. It is found that the flow stress depends not only on the dislocation density but also on the arrangement of dislocations. The flow stress varies linearly with dislocation density when the density is low, parabolically when the density is high, and finally insensitive to the density after complete cell formation. The temperature dependence of flow stress due to rearrangement within the tangles during deformation will be shown. The concept of frictional stress for group motion of dislocations, which is different from that for the motion of a single dislocation, will be introduced.

Cross Slip and Easy Glide: *D. H. Avery*, General Electric Co., and *W. A. Backofen*, Massachusetts Institute of Technology.

Intense primary and cross-slip traces were observed in easy glide on copper:6 pct aluminum single crystals deformed in tension. A mechanism of cooperative source operation is developed which recognizes that both the applied stress and that resolved from dislocation arrays contribute to the stress on a source. By considering the stress from arrays, the activation of secondary systems in easy glide and the difference in appearance of slip markings on pure and solid solution crystals are explained. Furthermore the cross-slip system is predicted to be second favored during easy glide for a large range of orientations.

Vacuum Techniques in Metallurgy

9:00 am to 12:00 m — Shelby Room

(Sponsored by EMD High Temperature Metals Committee)

Chairmen:

R. G. Jones, Wah Chang Corp., and *W. A. Krivsky*, Brush Beryllium Corp.

The Zone Purification of Yttrium: *W. C. Necker*, General Electric Co.

This paper will describe the equipment, process, and techniques used to maintain and traverse a molten zone thru metallic yttrium. The process includes high vacuum, high-frequency induction, and floating-zone processing.

It will include a brief discussion of zone refining mechanisms and their influences on choice of the process selected for zone refining of yttrium. The reasons for attempting the process will be mentioned.

The equipment design, including power source, coil design, vacuum system, and vacuum measurement will be discussed in detail. Slides showing equipment details will be available.

The preparation of specimens and the procedures followed in processing the specimens will be given. Phenomena observed during processing will be discussed.

The benefits obtained on yttrium as a result of processing will be limited to a slide showing a processed specimen, several metallographic pictures, and a mention of the changes in impurity levels and some mechanical properties.

The limitations and potential of the equipment with respect to other materials including the reactive metals will be discussed.

Separation of Sulphides by Vacuum Distillation: *G. N Banks, R. A. Campbell*, and *R. R. Rogers*, Canadian Department of Mines and Technical Surveys.

A laboratory investigation was made of the separation, by vacuum distillation, of lead and zinc compounds contained in complex sulphide ore concentrates. The effects of temperature, pressure, and heating time were studied. It was found that under suitable conditions, lead-free residues could be obtained from a lead-zinc-copper-iron concentrate, and also from a zinc concentrate. In both cases it was found that the zinc remaining in the residue could be removed by a second distillation at a higher temperature.

The Status of Vacuum Methods of Gas Analysis for Oxygen, Nitrogen, and Hydrogen in Metals: *N. A. Gokcen*, Aerospace Corp.

The improvements and limitations in current methods of analysis of oxygen, nitrogen, and hydrogen in metals and alloys by the vacuum technique, and the absolute accuracy of results are discussed. New data on the reliability of the author's technique are supplied. Methods of preparing highly accurate standard samples are described.

Brazing and Heat Treating in Vacuum: *R. F. Gunow*, and *C. Stitzlein*, Vac-Hyd Processing Corp.

New materials for the nuclear, aircraft, and rocket industries have led to the development of vacuum furnaces for brazing and heat treating processes. Two classes of furnaces have evolved: the *hot-wall* and the *cold-wall*.

Advantages of vacuum processing over the more familiar gas atmosphere processing are minimizing contamination and control over surface reactions. High-temperature alloys containing the refractory metals are susceptive to gas reactions that produce surface films which in turn prevent the wetting of brazing alloys. Also gases are absorbed in some metals, such as hydrogen in titanium, and have an adverse effect on physical properties. Vacuum processing alleviates these problems.

Many complicated shapes are now being produced with vacuum-brazing techniques. Brazing alloys suitable for use with vacuum make possible the production of fine corrugated high-temperature heat exchangers, turbine assemblies, and nuclear fuel elements.

New Developments in Oil Diffusion Pump Design for Vacuum Metallurgy: *M. H. Hablanian*, National Research Corp.

Significant improvements in the performance of diffusion pumps have been achieved in recent years. This paper presents latest developments in pump design and discusses them in reference to metallurgical applications. Methods of reducing the rate of backstreaming oil into the vacuum system and improvements in pumping speed, volume, forepressure tolerance, and ultimate pressure are discussed. Problems involving design of diffusion pumps and high-vacuum systems for metallurgical processing at pressures below 1×10^{-5} Torr (mm Hg) are considered. Specific examples are cited for the conventional vacuum resistance and sintering furnaces and for electron beam technology in melting, welding, and zone refining with particular application to refractory metals.

WEDNESDAY, OCTOBER 25

Oxidation and Corrosion

9:00 to 10:30 am — Sky Room

(Based on Submission of Research Abstracts)

Chairmen:

C. E. Birchenall, University of Delaware, and *D. V. Ragone*, University of Michigan.

Oxidation of 90Nb-10Ti Alloy in the Temperature Range 500-1200°C: *T. L. Kolski*, E. I. du Pont de Nemours and Co., Inc.

Reaction rates of 90Nb-10Ti alloy with oxygen at one atmosphere were determined gravimetrically at temperatures between 500° and 1200°C, for reaction times ranging from 25 to 200 hr. An anomalously high oxidation rate near 700°C follows a breakaway transition in kinetics after 15 to 20 hr from a rate which originally decreases with time. A mild breakaway transition occurs at 600°C after about 60 hr, but total oxygen absorption is low even after 150 hr. Oxidation rate at 500°C is extremely slow, total uptake of oxygen amounting to only 1.4 mg per sq cm after 200 hr. Above 700°C a rate inversion occurs with increasing temperature; oxidation rate passes through a minimum near 800°C, then rises with further temperature increase.

Analysis and X-ray diffraction patterns of outer scales formed on 90Nb-10Ti at 800°C or higher tentatively indicate that these consist of solid solutions of TiO_2 in Nb_2O_5. The outer scale formed at 700°C is not fully oxidized and exhibits a diffraction pattern different from the one characteristic of scales formed at higher temperatures. Little or no loose outer scale is formed at higher temperatures but the absorbed oxygen forms a hard case of tightly adherent oxide. Oxygen contaminated zones are formed under the outer oxide growth at higher temperatures, and attain thicknesses which increase markedly with oxidation temperature. A tentative mechanism for the accelerated oxidation observed near 700°C is proposed; it is based on severe surface cracking of the oxygen contaminated zone.

Oxidation of Cobalt in Carbon Monoxide-Carbon Dioxide Mixtures: *F. S. Pettit* and *J. B. Wagner, Jr.*, Yale University.

The oxidation kinetics of cobalt have been studied in pure carbon dioxide and carbon monoxide-carbon dioxide mixtures under a total pressure of 1 atm in the temperature range from 920° to 1200°C. After a short induction period, the oxidation rates were found to be linear and transformed to parabolic rates as the oxide approached thicknesses of about 5×10^{-3} cm.

The linear rates were found to be directly proportional to the mole fraction of carbon dioxide in the gas phase and the rate-determining step for this oxidaion process was the dissociation of carbon dioxide into carbon monoxide and adsorbed oxygen atoms or ions.

The parabolic rates, where a diffusion process was rate-determining, were found to be affected by the presence of porosity and voids in the oxide layer. The oxide formed during the linear and parabolic oxidation was composed entirely of cobaltous oxide (CoO), however, in some instances the oxide formed during parabolic oxidation consisted of a dense, crystalline, outer layer, and a porous, powdery, inner layer which indicated that these two structurally different oxide layers were formed by different oxidation mechanisms. In other cases only the dense, crystalline layer was observed. The parabolic data have been analyzed in terms of specimen geometry and purity, plasticity of the oxide and porosity of the oxide such that the occasional appearance of two structurally different layers in the oxide is explained.

This work was supported by the US Atomic Energy Commission.

Kinetics of Solution of Transition Elements in Several Liquid Metals: *D. B. Jugle* and *J. B. Darby, Jr.*, Argonne National Laboratory.

The processes by which liquid metals attack solid metals and alloys have been sought by many investigators in recent years. Most of the research has been inspired by the necessity for information related to a particular application and, in general, has been restricted to materials which have exceptional resistance to attack by liquid metals.

The purpose of this investigation was to explore systematically the rates of solution of several transition metals in pure liquid metals (99.99+ pct purity) under controlled conditions. The elements Ti, V, Cr, Fe, Co, and Ni were dissolved in liquid aluminum and liquid tin under a high vacuum at 700°, 750°, 800°C (aluminum), and 600°, 700°, 800°C (tin) while rotating the specimen at a constant velocity. In addition, the rate of solution was determined under similar conditions for some of these elements in liquid gallium. In all these experiments the concentrations of the solute in the liquid solvent were kept at very low levels compared with the solubility limits. A determination has been made of the liquidus curve in the temperature interval 550° to 850°C for six of the considered binary systems involving tin.

The corrosion [C (g dissolved per sq cm)] at a given temperature and in a given solvent was a linear function of time for all metals investigated. The rate obeyed an Arrhenius type equation, i.e., $K = K_o \exp -(Q/RT)$. For a series of solutes in the same solvent the rate of solution was related to the solubility limits at the temperature of the investigation.

This abstract is based on work performed at the Argonne National Laboratory under the auspices of the US Atomic Energy Commission.

Intergranular Corrosion of Aluminum in Hydrochloric Acid: *O. P. Arora*, and *Marvin Metzger*, University of Illinois, and *G. R. Ramagopal*, University of California.

The rates of intergranular and general corrosion were studied in high-purity aluminum and some single-phase dilute alloys with particular reference to the effects of copper content and acid strength. The rate of boundary attack was found to rise markedly with increasing copper content, as does general attack. For general corrosion the effect was shown in a previous investigation to arise from the autocatalytic acceleration of corrosion (at copper levels as low as 0.002 pct) due to the presence of copper in the corrosion product, so that rates much higher than the intrinsic ones are produced. The same explanation is proposed for the effect of copper content on boundary corrosion although this could not be studied in the same detail. The ratio of the intergranular to the general corrosion rate varied with acid composition from nearly unity to 50. Thus a selective intergranular-corrosion phenomenon corresponding to high values of this ratio is not a general characteristic of this system; it occurs under conditions where catalysis has a greater influence on boundary than on general corrosion and magnifies the intrinsic difference between the two. The addition of metallic ions to the acid yields weaker catalytic effects; in this case also, boundary and general corrosion are stimulated to different extents.

This work was supported by the Office of Naval Research.

Reactivity of Tungsten With High Temperature Materials in the Range 4000-6000°F: *F. J. Lally* and *R. H. Hiltz*, Thompson Ramo Wooldridge Inc.

The increasing application of tungsten in very high-temperature equipment has been hampered by the lack of knowledge of its compatibility with other materials of construction at temperature above 4000°F. Prime interest is centered at present on the use of tungsten as a non-eroding throat material in uncooled, solid-propellant rocket nozzles. This program was initiated to investigate the compatibility or reactivity of tungsten with a variety of nozzle structural materials over the range of temperatures, 4000° to 6000°F, and times up to 120 sec. These materials were the high-melting oxides and carbides, graphite, and phenolic resins.

The results of this study show that tungsten reacts with all of these materials. If reactions are confined to the solid state alone they are not detrimental and could be tolerated in a structure for short periods of time. If a liquid phase is involved, however, due either to melting of one of the reactants or the formation of a liquid as a reaction product, structural integrity is destroyed.

Nuclear Fuel Reprocessing II: New Foreign and American Developments

2:00 to 5:00 pm — Terrace Room

(Sponsored by EMD Nuclear Fuel Reprocessing Committee)

Chairmen:

J. A. Leary, Los Alamos Scientific Laboratory, and *G. E. Brand*, Atomics International.

WEDNESDAY, OCTOBER 25

Thorium Recycle Development: *D. E. Ferguson, E. D. Arnold, O. C. Dean,* and *W. S. Ernst, Jr.,* Oak Ridge National Laboratory.

Improved methods are being developed at Oak Ridge National Laboratory for the preparation and fabrication of ThO_2 fuels. The activity due to the decay products of U-232 in U-233 and Th-228 in thorium from power reactors necessitates preparation and fabrication of new fuel elements from recycled fuel by remote methods. Pure ThO_2 or mixed ThO_2-UO_2 may be prepared by the sol-gel process by simple steps that can be easily operated behind shielding and loaded into fuel element tubes by vibratory compaction. A thoria hydrosol is evaporated to produce a gel which, when calcined to only 1150°C, yields large ThO_2 particles of greater than 99 pct of theoretical density. The addition of uranium to the sol and an additional calcination at 1150°C in a hydrogen-containing atmosphere produces a homogeneous solid solution of ThO_2-UO_2. ThO_2-4 pct UO_2 prepared on a 1-kg scale was consistently compacted with simple, inexpensive pneumatic vibrators to 8.7 g per cu cm, 87 pct of theoretical density, in 1/4-x 11-in. stainless steel irradiation capsules. Eight of these capsules, now being irradiated in the NRX, will be discharged and examined after reaching a level of 15,000 Mwd per ton. With ThO_2-UO_2 prepared by arc fusion, densities as high as 9.0 g per cu cm were obtained in 1/2 in.-diam tubes by vibratory compaction. The density of oxide fuel loaded by this method varied about ±0.06 g per cu cm in single tubes and multitube bundles were loaded with no greater variation from one tube to another.

Scientific Development Connected to the Eurochemic Reprocessing Project: *R. Rometsch,* Eurochemic (Belgium).

The characteristics of the Eurochemic processing plant reflect the summing up and compromising over many different demands. The plant will have to prove the feasibility of multi-purpose reprocessing by accepting a wide range of various fuel elements; it must be conceived as a pilot plant, but should guarantee a certain production capacity and, last but not least, has to provide economic data with sufficient precision to allow extrapolation to a similar high capacity plant.

To try out new chemical processes on top of these demands, seemed excluded. Hence, the well known Purex process was adopted as a basic flowsheet. Nevertheless, the research tasks connected to the design work remain numerous and exciting.

Three groups of problems will be discussed in some detail:

1) collection of flowsheet data for a multi-purpose dissolver in laboratory experiments and test operation of a glass dissolver, about 1/3 of the size of the plant dissolver;
2) extraction flowsheet adaptions and their checking in laboratory mixer-settler-batteries, particularly regarding decontamination factor in the first cycle and the separation of plutonium; and
3) comparison of established, proposed and new methods for final plutonium purification.

Most of the experimental work is still going on and will continue during construction and running in of the plant.

Reprocessing Fast Reactor Fuels at Dounreay: *D. M. Donaldson, K. Hartley, P. Lees,* and *N. Parkinson,* Atomic Energy Authority (United Kingdom).

The development of a fast breeder reactor system fuelled (sic) with plutonium is an important part of the United Kingdom's work on advanced power reactors, fuels under consideration including oxides, cermets, and carbides. The wide range of types of fuels and probable compositions makes it essential to establish basic technologies for processing any fuels that may be irradiated. Dounreay re-processing work has been directed towards *head-end* processes for the preparation of fuel solutions which can be handled either by the pilot processing plant or the main Dounreay Fast Reactor Chemical Re-processing plant and to a study of the special problems arising from the solvent extraction processing of high burn-up fuels containing 15 to 50 pct Pu, in tributyl phosphate-nitric acid systems.

The driving charge of the Dounreay Fast Reactor, which also provides an irradiation facility for experimental fuels and sub-assemblies, is an enriched uranium-molybdenum alloy, the dissolution of which in nitric acid presents solubility and waste storage problems: controlled oxidation of such alloys followed by sublimation and trapping of the molybdic oxide offers a useful *head-end* treatment for molybdenum removal as well as providing some decontamination from ruthenium. Dissolution of carbides in nitric acid produces appreciable quantities of mellitic acid: the presence of organic materials in raffinates which are evaporated presents a potential hazard, so that hydrolysis or controlled oxidation of carbides may be necessary. The total dissolution of stainless steel-oxide cermets results in considerable volumes of high active wastes and a mechanical head-end process combined with leaching offers considerable potential advantage if a nitric acid soluble fissile phase is used. Solid solutions of mixed oxides of uranium and plutonium containing not more than about 40 pct PuO_2 are completely soluble in nitric acid without fluoride additions. The Darex process also appears applicable to such fuels, but for materials requiring fluoride additions a preliminary size reduction followed by a *Niflex*-type dissolution may offer a more satisfactory approach, although the corrosion of plant materials then requires special study.

The relationships governing the partition of macro-quantities of uranium and plutonium in the nitric acid, tributyl phosphate-odorless (sic) kerosene system have been established together with the solubilities of the TBP solvates of tetravalent and hexavalent plutonium. The conditioning of the various valency states of plutonium for adjustment of extraction properties in the TBP-nitric acid system presents more difficult problems than in a natural uranium system containing micro-quantities of plutonium. Similarly, acid butyl phosphates resulting from solvent radiolysis present problems in the retention of plutonium and in the solubility of their metallic salts. The effect of some of these problems on the TBP process have been studied.

Recent Developments in the Processing of Nuclear Fuel by Ion Exchange: *D. B. James* and *E. L. Christensen,* Los Alamos Scientific Laboratory.

Ion-exchange processes for thorium, uranium, and plutonium are reviewed as examples of ion exchange as a unit operation. A qualitative discussion of the various phenomena in both the solution phase and the resin phase which are used as driving forces to effect separations is presented.

Quadravalent thorium has a high affinity for cation-exchange resin and can be sorbed under conditions where lower-valent species are not sorbed. While thorium may only be partially complexed by the nitrate ion, the high affinity of the hexanitrate complex for anion resin results in strong sorption from concentrated nitrate solutions. Thorium is not sorbed from concentrated chloride solutions where many other metallic ions form sorbable anionic complexes.

The uranyl ion forms an anionic sulfate complex in dilute sulfate which allows it to be sorbed on anion-exchange resin or to be desorbed from cation-exchange resin. Sorbable anionic complexes form in both strong chloride and nitrate solutions.

The sorption of plutonium (III) on cation-exchange resin is used as a method of concentration and for changing the associated anion. Plutonium (IV) is strongly sorbed by anion-exchange resin from both strong chloride and nitrate solutions while plutonium (III) is not. Therefore, by proper valence adjustments plutonium can first be separated from those elements sorbed by anion-exchange resin and then from those which are not sorbed.

Corrosion of Containment Materials by Molten Zinc and Cadmium Alloys: *J. DeKany, H. Lavendel,* and *L. Burris,* Argonne National Laboratory.

A corrosion program was undertaken to find suitable container materials for the various liquid metal systems encountered during the development of pyrometallurgical processes for the recovery of uranium and plutonium from spent reactor fuels. The liquid metals under consideration were zinc, cadmium, and magnesium, either alone or in various combinations with each other.

The corrosion program consisted of 100-hr static capsule tests designed to screen a large number of materials. Promising materials were then subjected to 1000-hr dynamic capsule tests, or used in forced convection or thermal convection loops.

These tests have shown that molten cadmium alloys may be satisfactorily contained in mild steel or nickel-free ferrous alloys under static and dynamic conditions up to at least 550°C, and probably somewhat higher. At 700°C, however, there is intergranular attack of these steels by molten cadmium.

Few materials are resistant to molten zinc, at temperatures up to 800°C. Of the metals, only tungsten, tantalum, and molybdenum showed substantial resistance to attack by zinc. Tungsten was completely inert to the zinc system. Tantalum, which may be placed next in order of resistance, did undergo some attack after 100 hr at 750°C to depths of about 1 mil, through formation of a suspected intermetallic compound. Molybdenum was attacked to a slightly greater extent than tantalum by a solution process.

Alumina, silicon carbide, silicon nitride, zirconia, Vycor, and graphite were unaffected by zinc at 750°C. When magnesium was present in the zinc, Vycor and silicon nitride were severely corroded.

WEDNESDAY, OCTOBER 25

Corrosion of Materials of Construction for Multipurpose Head-end Equipment in Proposed Fuel Reprocessing Schemes: *W. E. Clark*, Oak Ridge National Laboratory.

The necessity for reprocessing a wide variety of experimental nuclear fuels has created interest in the development of multipurpose processing schemes. The possibility of handling both stainless steel and zirconium fuel types in the same process equipment is particularly attractive. This paper considers in some detail the corrosion problems in multipurpose aqueous head-end processes for these fuel types and compares them briefly with those in certain combustion and fused salt processes.

Dissolution of stainless steel fuels in aqua regia (Darex process) can be combined with dissolution of zirconium fuels in 1) NH_4F-H_2O_2 mixtures which can be contained in high cobalt alloy equipment or 2) carefully controlled nitrate-HF mixtures which appear to be containable in titanium. Alternatively Darex can be combined with a gas-solid combustion process for zirconium and niobium alloys by using a separate reactor (nickel alloy) for the latter and employing common equipment for feed adjustment and solvent extraction. Yet another alternative is to combine sulfuric acid dissolution of stainless steel fuels with NH_4F dissolution of zirconium fuels in a dissolver constructed of Ni-o-nel or similar alloys. Corrosion data for these proposed processes indicate that rates $\leq$ 3 mils per month may be attainable in all cases if process conditions can be controlled to the required degree.

Corrosion data are presented for competitive aqueous processes and are compared with those for the fused salt-volatility process. Areas requiring future corrosion work are outlined.

Diffusion

2:00 to 5:00 pm **Ballroom**

(Based on Submission of Research Abstracts)

Chairmen:

R. Baluffi, University of Illinois, and *H. I. Aaronson*, Ford Motor Co.

Thermal Diffusion of Vacancies in Pure Gold: *P. G. Shewmon*, Carnegie Institute of Technology.

The redistribution of solute that occurs in an initially homogeneous alloy upon annealing in a temperature gradient is called thermal diffusion. In the case of pure gold, the two diffusing species are gold atoms and vacancies. Thus this case will differ from a conventional alloy in that a time invariant vacancy concentration gradient will be maintained. Any flux of atoms which results will appear as a relative motion of markers placed in the wire. In the present work, pure gold wires were held in a temperature gradient of 270°C per cm. Marker movement occurred indicating a net flux of atoms toward the hot end. When the effect of the vacancy concentration gradient is subtracted out, the heat of transport of the gold atoms, Q^*, is found to be about 15 kcal per mol. This indicates that in the absence of the vacancy concentration gradient, the jumps of the atoms would be biased toward the cold end of the piece.

Previous workers have looked for a marker movement of this sort in Zn and Fe, but have found none. Work on silver is in progress and will be reported on.

This work was supported by the Office of Naval Research.

Interstitial Diffusion in Dilute Ternary Austenites: *J. S. Kirkaldy* and *G. R. Purdy*, McMaster University.

For the iron-rich ternary austenite in which the interstitial carbon solute is very much more mobile than another substitutional solute the Onsager reciprocal relations simplify to

$$D_{11}\frac{\partial \mu_1}{\partial N_2} = D_{12}\frac{\partial \mu_1}{\partial N_1}.$$

When this is combined with a nearest neighbour calculation of the thermodynamic activity of carbon in dilute solution

$$a_1 = N_1\left\{1 + \left(\frac{12\epsilon_{11}}{kT} + 2\right)N_1\right\}\left\{1 + 6\left(\frac{\epsilon_{12} - \epsilon_{31}}{kT}\right)N_2\right\},$$

one obtains the expression relating the off-diagonal coefficient, D_{12}, to the on-diagonal coefficient, D_{11},

$$\frac{D_{11}}{D_{12}} \cong 6\frac{(\epsilon_{12} - \epsilon_{31})}{kT}N_1.$$

Direct experimental measurements of this ratio for the systems Fe-C-Si, Fe-C-Mn and Fe-C-Ni compare favorably with the thermodynamic predictions obtained by using independent thermodynamic determinations of the coefficients involving the interaction energies, ϵ. This work was supported by grants from the National Research Council of Canada and the American Iron and Steel Institute.

Simulation of Banding in Steels: *R. J. Brigham, J. von Destinon-Forstmann*, and *J. S. Kirkaldy*, McMaster University.

Three-layer diffusion couples have been constructed so as to simulate the compositional variations which occur in commercial mild steels as a result of interdendritic segregation. It has been possible to quantitatively examine the banding behavior in the ternary systems Fe-Si-C, Fe-Mn-C, Fe-Ni-C, Fe-Cr-C, and Fe-P-C both as to the intensity of carbon segregation which occurs in the soaking (austenite) range and during the subsequent $\gamma \rightarrow \alpha$ transformation. The results confirm that the segregation of carbon which occurs before transformation is due to the equalization of the carbon activities. As expected from thermodynamic data, silicon, nickel and phosphorus-rich regions reject carbon while manganese and chromium-rich regions attract it. While this initial segregation is weak, in all cases but nickel it intensifies in the approach to and during transformation with the help of differential nucleation rates. In nickel couples the segregation direction reverses during transformation so that the pearlite-rich regions appear with the nickel. This emphasizes the predominant effect of that preferential nucleation which arises within a manganese-like constitution. This work was supported by a grant from the American Iron and Steel Institute.

Self-Diffusion in Liquid Tin: *R. A. Swalin* and *C. H. Ma*, University of Minnesota.

The self diffusion of tin-113 in tin has been investigated over a wide temperature interval (250° to 1000°C) in order to examine the general validity of the fluctuation theory of diffusion proposed by one of the authors.

The capillary-reservoir technique was used in the investigation. The capillaries were made of fused silica and had lengths of about 2 cm and inside diameters of 0.5 or 1.0 mm. During the diffusion-anneal treatment, the top of the capillaries were kept about 1°C hotter than the bottom in order to minimize convection. After the diffusion treatment, the capillaries were sectioned into wafers about 2 mm thick and the concentration of γ emitting tin-113 in each wafer was determined by use of a well-type scintillation counter in conjunction with a scaler and pulse height analyser. The diffusion constant was determined by the appropriate solution of Fick's second law. Upon plotting the data as the logarithm of the diffusion constant versus the reciprocal of absolute temperature, considerable positive curvature was found to occur at elevated temperatures. The data are in good agreement with the data obtained at low temperatures by Careri *et al* which were obtained by a different experimental arrangement.

Quantitatively, the fluctuation theory predicts that the self-diffusion coefficient of tin is given as a function of temperature by

$$D = 1.09 \times 10^{-10}\,T^2\ cm^2/sec$$

The theory predicts the magnitude of D quite well but the observed experimental temperature dependence is somewhat larger than T^2. Similar curvature has been reported by Meyer for mercury and Lodding for indium and the authors believe that the general features of the fluctuation model are proved to be valid.

Research sponsored by the Atomic Energy Commission.

Diffusion of Sulfur in Single Crystals of Lead Sulfide as a Function of Stoichiometry: *Martin Seltzer* and *J. B. Wagner, Jr.*, Yale University.

The diffusion of radioactive sulfur (S-35) into single crystals of lead sulfide has been studied as a function of deviation from stoichiometry, and defect concentration introduced by doping, in the temperature range 773° to 1023°K, in order to determine the predominant mechanism by which the anion diffuses in this compound. Measurements have been made using crystals of stoichiometric composition, and on others with 10^{15} excess sulfur atoms per cu cm or 10^{18} excess lead atoms per cu cm. The sulfur excess specimens showed the highest diffusivities in this tempera-

WEDNESDAY, OCTOBER 25

ture range, whereas lowest diffusion coefficients were obtained from the lead excess specimens. The data can be described by Arrhenius type equations to give an activation energy for diffusion of 1.38 ev for the stoichiometric material, while values of 1.16 and 1.22 ev were obtained for the lead excess and sulfur excess crystals, respectively.

Diffusion measurements have also been performed on specimens doped with 0.5 or 0.05 mole pct Ag_2S or Bi_2S_3, which were annealed under the same conditions as those required to obtain undoped stoichiometric PbS crystals.

These data appear to rule out the possibility that the negative ion diffuses by jumping into isolated negative ion vacancies. The importance of neutral vacancy pairs as a mode for anion diffusion is considered.

This work was supported by the Office of Naval Research.

Diffusion of Al^{26} and Mn^{54} in Aluminum: *T. S. Lundy* and *J. F. Murdock*, Oak Ridge National Laboratory.

The diffusion coefficients of Al^{26} and Mn^{54} in high-purity, large-grained aluminum have been measured at 15° intervals from 450° to 650°C. Thin disks of aluminum containing radioactivities produced by bombardment with 22 mev protons in the 86-in. ORNL Cyclotron were placed in contact with polished flat faces of *infinitely thick*, inactive cylinders. The assemblies were held together at heat-treating temperatures in metal capsules making use of the large thermal expansion of aluminum. Isothermal annealing, precision lathe sectioning, and counting of the gamma activities of both Al^{26} and Mn^{54} were performed by standard techniques.

The applicable solution to Fick's second law for specimens having the described geometry is

$$\frac{A(x)}{A_0} = \frac{1}{2}\left\{\operatorname{erf}\frac{h-x}{2\sqrt{Dt}} + \operatorname{erf}\frac{h+x}{2\sqrt{Dt}}\right\}$$

where A(x) is the activity at a normal distance x from the specimen surface

A_0 is the original disk activity
h is the disk thickness
D is the diffusion coefficient
t is the time of heat treatment at temperature T

The experimental data were plotted as ln A(x) versus x^2 and the slopes of the resulting straight lines were set equal to the following equation, which is the derivative of the logarithm of first equation with respect to x^2 evaluated at $x = h$:

$$\left(\frac{d \ln A(x)}{d x^2}\right)_{x=h} = \frac{\frac{h^2}{e^{Dt}} - 1}{2h\sqrt{\pi Dt}\operatorname{erf}\frac{h}{\sqrt{Dt}}}$$

Diffusion coefficients were then calculated using a simple iterative process.

Unexpectedly large values of A(x) near the specimen surface may be explained by the presence of a layer of Al_2O_3 at the disk-inactive cylinder interface. This layer acts as a larger barrier for the diffusion of Mn^{54} than for that of the Al^{26m}.

Plots of ln D versus 1/T were found to fit the following equations:

$$D_{Al}26m = 1.3\, e^{-\frac{28,000}{RT}}$$

$$D_{Mn}54 = 3.2\, e^{-\frac{28,500}{RT}}$$

Diffusional Contribution to the Knudsen Cell Effusion Flux: *W. L. Winterbottom* and *J. P. Hirth*, Carnegie Institute of Technology.

It has been recognized for some time that the geometry of a Knudsen vapor effusion cell influences the kinetics of the molecular effusion of the vapor from within the cell. Clausing, Whitman and Motzfeldt have independently treated several aspects of this problem and have given treatments which allow for the correction of the effusion flux to account for these factors so that the flux obtained will reflect the actual vapor pressure within the cell.

However, no treatment has been reported of the contribution to the effusion flux made by the surface diffusion out through the orifice of the effusate adsorbed on the walls within the cell. Treating the steady-state kinetics for both surface diffusion and vapor effusion out through the orifice the applicable differential equations for this model have been solved approximately. Although the required thermodynamic data is sparse, the derived relations indicate the diffusion flux for the orifice dimensions commonly employed may contribute an appreciable fraction of the total weight loss of the cell during an effusion experiment. If not corrected for this diffusional contribution would yield equilibrium vapor pressures in excess of the actual values.

Phase Relationships and Thermodynamics

2:00 to 5:00 pm — **Shelby Room**

(Based on Submission of Research Abstracts)

Chairman:
M. V. Nevitt, Argonne National Laboratories

Phase Relationships in the Ti-Mn System: *R. M. Waterstrat, B. N. Das*, and *P. A. Beck*, University of Illinois.

A metallographic and X-ray diffraction study was carried out with alloys arc-melted from iodide Ti and hydrogen-annealed electrolytic Mn, homogenized at 900° to 1175°C. The following intermediate phases were found: $(TiMn_4)R$, $TiMn_3$, Laves phase $TiMn_2$ to near TiMn, $(TiMn)\phi$ and $(TiMn)\theta$. Ti is soluble in α-Mn and stabilizes it up to about 1150°C at 10 at pct Ti.

Ternary Laves Phase With Transition Elements and Silicon: *D. I. Bardos, K. P. Gupta*, and *P. A. Beck*, University of Illinois

Combinations of A-elements from the Sc-, Ti-, V-, and Cr-groups with B-elements Fe, Co, and Ni form binary Laves phases, AB_2, when the average number of electrons outside the closed shells is less than eight. When this number is eight, or larger, no binary Laves phase is formed. It was found, however, that the missing Laves phases become stabilized on substituting silicon for about ⅛ to ⅔ of the B-element. The following alloys were found to consist largely of $MgZn_2$-type Laves phase at 1100° or 1200°C: Ti_2Ni_3Si, $V_4Ni_5Si_3$, Cb_2Ni_3Si, Ta_2Ni_3Si, Mo_2Ni_3Si, W_2Ni_3Si, $V_4Co_5Si_3$, Mo_2Co_3Si, and W_2Co_3Si.

Lattice Spacing Relationships in H.C.P. ε and η Phases in Alloys of Zn and Cd with the Noble Metals: *T. B. Massalski* and *H. W. King*, Mellon Institute.

The lattice spacings of hcp ε and η phases in the systems of Cu, Ag, Au with Zn, and in Ag-Cd have been studied by the X-ray powder method.

The results show consistent trends in the changes of axial ratio and lattice spacings when plotted as functions of electron:atom ratio. The observed trends follow the same general pattern as those reported earlier for hcp ζ phases and can be interpreted in terms of distortions of the Brillouin zone following interactions between the Fermi surface and various zone faces. The results for the ε phases indicate that, in addition to the overlap of Fermi electrons across the {10.0} faces of the Brillouin zone, overlap also occurs across the {00.2} faces at $c/a \cong 1.85$ and that to a first approximation this value is independent of the noble metal alloyed with Zn or Cd.

Comparison of the trends observed in the ε phases with similar trends in the hcp ζ phases in the systems Ag-Al, Ag-Sn, and Ag-Sb suggests that overlap of the Fermi electrons across the {00.2} faces of the Brillouin zone may occur at lower values of electron concentration when the noble metals are alloyed with elements other than Zn or Cd.

The theory of the ε and η phases proposed by Jones and the general shape of the ε and η phase fields are discussed in relation to the two types of Brillouin zone overlap.

Phase Diagram of the Pseudo-Binary System UAl_2-UFe_2: *D. J. Lam, J. B. Darby, Jr.*, and *J. W. Downey*, Argonne National Laboratory.

The approximate phase boundaries for the pseudo-binary system UAl_2-UFe_2 have been investigated using optical and X-ray metallography. Both components have the C-15 structure, but they exhibit limited miscibility in each other. Two intermediate phases exist in the system. Their structures have not been identified.

This abstract is based on work performed at the Argonne National Laboratory under the auspices of the US Atomic Energy Commission.

WEDNESDAY, OCTOBER 25

An X-Ray Investigation of Alloys of Technetium-99 With Some Transition Elements of the First and Third Long Periods:

J. B. Darby, Jr., D. J. Lam, J. W. Downey, and *L. J. Norton,* Argonne National Laboratory.

Little information is available on constitution diagrams involving technetium since this element does not occur naturally. It can be produced, however, as a fission product in the reprocessing of nuclear fuel elements. Recently sufficient quantities of the stable isotope technetium-99 have become available to permit the use of conventional techniques associated with phase diagram studies of radioactive materials.

An extensive investigation is in progress to determine the solid solution ranges and the occurrence of intermetallic compounds in binary systems with technetium-99 as a component and a transition element of the first or third long periods as the other component. Arc-melted buttons, having a mass of the order of one gram, are prepared starting with a composition at the technetium-rich end of a given system. Subsequent alloys, the number of which depends on the complexity of the phase relations, are prepared by dilution in a step-wise manner with the second component. The resulting structures are examined by x-ray metallography.

The results to date indicate that technetium behaves very much like the other members of the Mn group when alloyed with transition elements. For example, the α-Mn structure (Type A-12) exists in the Sc-Tc and Ti-Tc systems and V and Fe have substantial solid solubility in technetium.

This abstract is based on work performed at the Argonne National Laboratory under the auspices of the US Atomic Energy Commission.

Heat Capacity of α-Phase Cu-Zn Alloys:

M. C. Huffstutler, Jr., and *R. R. Hultgren,* University of California.

The heat capacities of three Cu-Zn alloys in the composition range 10 to 35 wt pct Zn have been measured, by an isothermal technique, over the range 13° to 300°K. These data are used in a study of the effect of alloying on low-temperature C_p values and the thermal entropy obtained by integration of $C_p \frac{dT}{T}$.

The general implications of this work relative to the empirical correlation of heat capacities and entropies of formation of similar alloys have been considered.

Solubility and Precipitation of Boron Nitride in Iron-Boron Alloys:

R. W. Fountain, Union Carbide Metals Co., and *John Chipman,* Massachusetts Institute of Technology.

Using a Sieverts' type apparatus for determining the solubility of nitrogen in iron-boron alloys, a study has been made of the conditions under which BN forms from dissolved boron and nitrogen and from dissolved nitrogen and the compound Fe_2B. From these results, the two-phase equilibrium between γ iron and BN is established and also the three-phase equilibrium between γ iron, BN, and Fe_2B in the temperature range of 950° to 1150°C. The above equilibria are calculated for alpha iron from the measured solubility relations.

Under the conditions of these experiments, the BN was found to precipitate as thin flakes whose lack of three-dimension crystal perfection is observed by absence of certain reflections in the X-ray diffraction patterns of extracted residues.

The following thermodynamic properties of the system have been determined in the range of 950° to 1150°C.:

1) The effect of boron on the solubility and activity coefficient of nitrogen in γ iron;

2) The equilibrium constant, free energy, and heat of formation of BN from dissolved boron and gaseous nitrogen;

3) The equilibrium constant, free energy, and heat of formation of BN from Fe_2B and gaseous nitrogen;

4) The equilibrium constant, free energy, and heat of formation of BN from Fe_2B and dissolved nitrogen; and

5) The free energy of solution of boron in γ iron and the free energy of formation of Fe_2B.

Boron has a greater ability to remove nitrogen from solution than either aluminum or vanadium. This ability is restricted by the limited solubility of Fe_2B.

The application of these results to the influence of boron on the resistance of low-carbon steel to strain aging and the hardenability of heat-treatable steels is discussed.

Some Observations on the U + Nb — H System:

O. M. Katz and *E. A. Gulbransen,* Westinghouse Electric Corp.

The reaction of bcc gamma phase U + 10 wt pct Nb and U + 90 wt pct Nb with hydrogen was investigated below one atmosphere of pressure between 264° and 571°C. Dissociation pressure hysteresis was found in the 264°C isotherm for the U + 10 wt pct Nb alloy, but did not exist at 299°C. The maximum composition reached at 264°C was $U\,Nb_{0.307}\,H_{1.588}$. Even though UO_2 was present on the sample, it was felt that the hysteresis was an intrinsic property of the metal lattice. The formation of a reaction product on quenching in U + 10 wt pct Nb-H alloys indicated a low temperature phase which could have formed by a martensitic mechanism.

No hysteresis was found in the equilibrium isotherms for U + 90 wt pct Nb-H alloys. The relative partial molar enthalpies and entropies decreased from a mole ratio, n_H/n_{U+Nb}, of 0.04 to 0.40 and then increased to $n_H/n_{U+Nb} = 0.575$. Relative partial molar free energies increased with increasing composition at a given temperature and increasing temperature at a given composition. A given percentage of hydrogen was less stable in a U + 90 wt pct Nb alloy than in pure Nb.

Thermodynamic Properties of Solid Copper-Nickel Alloys:

R. A. Rapp, Wright Patterson Air Force Base, and *Fritz Maak,* Carnegie Institute of Technology.

The following galvanic cell with the solid electrolyte (0.85 ZrO_2 + 0.15 CaO) was used to determine the thermodynamic activity of nickel at 700° and 1000°C in Cu-Ni alloys containing 10 to 90 at pct Ni:

Pt|Ni, NiO|(0.85 ZrO_2 + 0.15 CaO)|Cu-Ni alloy, NiO|NiO|Pt.

The activity of nickel in the Cu-Ni alloy may be related to the measured equilibrium emf E of the cell by the expression

$$\ln a_{Ni\ (alloy)} = -2\,EF/RT.$$

The activities of copper and nickel in Cu-Ni alloys exhibited positive deviations from Raoult's law. The system is characterized by positive enthalpies and small negative entropies of mixing. The change in electronic configuration upon alloying copper with nickel was indicated by only minor effects in the thermodynamic functions at these temperatures. Tentative calculations involving ΔH data from 640°C and ΔS data from 850°C indicate that a miscibility gap should occur at about 300°C and be shaped similarly to that in the Au-Ni system.

The use of this galvanic cell technique involving the solid electrolyte (0.85 ZrO_2 + 0.15 CaO) is relatively fast, simple, and requires no complicated apparatus. The cell may be used at high temperatures, from about 700°C until either a component of the alloy or an oxide exhibits an appreciable vapor pressure. In principle, the technique is applicable to an alloy A-B without the occurrence of a displacement reaction if $|\Delta G^\circ_{ZrOs}| >> |\Delta G_{AO}| > |\Delta G_{BO}|$ within the cell. However, the solution of very active alloying metals into ZrO_2 at very high temperatures could introduce significant excess electron conduction into the electrolyte. The use of a neutral atmosphere with the cell is decisive for the elimination of side reactions between the cell and the gas.

Financial support from a Fulbright scholarship is acknowledged.

Thermodynamic Properties of the Columbium-Nitrogen Terminal Solid Solution:

J. R. Cost and *C. A. Wert,* University of Illinois.

When a diatomic gas goes into solution ideally in a metal, Sievert's Law, (i.e. that the concentration is proportional to the square root of the partial pressure of the gas,) is obeyed and also the heat of solution is a constant. The temperature-pressure-composition equilibrium was studied for the Cb-N system to determine to what extent deviations from ideality were present. Only small deviations were found and these are discussed. Two experimental methods of obtaining this data were used and found to be in good agreement. Both of these methods also gave information as to the terminal solubility of nitrogen in the columbium alpha solid solution as well as the decomposition pressure of Cb_2N, the phase which is in equilibrium with the saturated solid solution. The data for this system is analyzed in terms of an idealized gas-metal system and a simplified phase diagram for pressure, temperature, and composition is proposed which demonstrates how the sum of the enthalpies of the first two of the following relations equals that of the third.

$$\tfrac{1}{2}\,N_{2(g)} = N \text{ (in solution in Cb)}$$
$$N \text{ (in solution in Cb)} + 2\,Cb_{(s)} = Cb_2N_{(s)}$$
$$\tfrac{1}{2}\,N_{2(g)} + 2\,Cb_{(s)} = Cb_2N_{(s)}$$

WEDNESDAY, OCTOBER 25

Standard enthalpies, entropies, and free energies for the above reactions are given and discussed.

Financial assistance for this research was received from the Air Force and the Atomic Energy Commission under contract numbers AF 49(638) 672 and AEC AT(11-1) 67 Niobium. One of the authors (J.R.C.) was holder of a Union Carbide Research Fellowship.

Deformation I

2:00 to 5:00 pm — **Sky Room**

(Based on Submission of Research Abstracts)

Chairmen:

G. F. Bolling, Westinghouse Electric Corp., and *J. O. Brittain*, Northwestern University.

Effects of Thermal-Mechanical Treatments on Faulting in Some F.C.C. Alloys: *D. E. Mikkola* and *J. B. Cohen*, Northwestern University.

Deformation twin faults were found in Cu_3Au and Cu-14.3 at pct Al alloys deformed at room temperature as well as at low temperatures; the concentration of both twin and stacking faults increased with decreasing temperatures of cold-work. The ratio of twin to stacking faults was about two in both alloys after deformation below room temperature; this suggests that the mechanisms for the two faults are associated.

An increase in fault probability associated with a solute of a different valence but the same size as the solvent was found by comparing deformed Ag and Ag 6 at pct Al. The twin and stacking fault probability was greater in Cu_3Au than in copper or gold, although the atoms differ in size only. Furthermore, the stacking fault probabilities were slightly larger in slow cooled specimens of Cu_3Au and Cu-14.3 at pct Al (high order) than in quenched specimens. Measured fault probabilities are closely related to the measured fault energies in alloys as well as pure metals; the observed results can be interpreted in terms of electronic effects on alloying or ordering.

On heating filings of the Cu-14.3 at pct Al alloy, stacking faults annealed out in two stages. The first stage was characterized by a decrease in fault probability after a short time at temperature, after which no change was observed for long times. This decrease in probability increased with increasing temperature. Most of the faults annealed out in the second stage; large changes in hardness and micro-strain indicated that this latter stage was associated with recrystallization. The activation energy for the second stage was 64 ± 5 kcal per gm atom, in good agreement with the activation energy for interdiffusion in this system. Twin faults were more stable than stacking faults.

This research was sponsored by ONR and by ARPA (Contract No. SD-67).

Influence of Grain Size and Specimen Size on Unidirectional Polycrystal Plasticity: *R. W. Armstrong*, Westinghouse Electric Corp.

Two cumulative *size* effects are responsible for the increased strength of a polycrystalline aggregate above the single crystal strength at temperatures and strain rates where diffusion does not play a large part in the deformation: a *specimen size* effect and a *grain size* effect. The two effects produce strengthening of a material for different reasons.

The *specimen size* effect occurs when few grains are present in a specimen cross-section (say less than 20), and this effect is due mainly to the orientation dependence of crystal plastic flow. This effect on the flow stress of an aggregate is derived from two closely related and crystal orientation dependent considerations: 1) the critical resolved shear stress must be achieved on the slip systems of all the grains contributing to bulk plastic flow; and, 2) the choice of slip systems in adjacent grains of a polycrystal is affected by the relative orientation of the grains to each other, as well as to the applied stress, because strain continuity must be maintained at the grain boundaries. As a result of this *size* effect, the specimen flow stress, σ_f, can vary in a range from twice the single crystal critical resolved shear stress, τ_c (for a *bamboo* structure containing at least one grain of appropriate orientation), through the average orientation factor, m_o, times the single crystal strength as described by Sachs (1928), to the grain accommodation factor, m, times the multislip single crystal strength, τ_m, as described by Taylor (1956) and Kocks (1958, 1960).

$$2\tau_c \leq \sigma_f \leq m\,\tau_m$$

The *grain size* effect occurs in polycrystalline materials because grain boundaries are obstacles to the initiation of bulk yielding and subsequent propagation of plastic flow, and these boundaries require internal concentrations of stress proportional to the grain diameters from within the grain volumes to induce general yielding of the aggregate. The *grain size* effect comes into play when sufficient grain boundaries are present in the specimen volume to allow measurement of the combined effects of the statistical orientation constraints on plastic flow and the average grain boundary barrier to flow from within the grains. This effect explains values of the flow stress greater than $m\,\tau_m$ and it results in an explicit dependence of the flow stress on grain size, l:

$$\sigma_f = m\,\tau_m + mk_s l^{-1/2}$$

The parameter, k_s, depends on the number of deformation systems and the stress required to cause plastic flow on them in the vicinity of a grain boundary. k_s is affected in two ways by impurities in a material:

1) The number of bulk deformation systems may be altered (slip and twinning behavior), and, 2) the pinning of dislocations may occur.

Part of this research was conducted under the direction of Professor N. J. Petch and with support from the United Kingdom Department of Scientific and Industrial Research.

Grain-Size Effects on the Deformation of Tantalum at Low Temperatures: *R. C. Koo*, Westinghouse Electric Corp.

The variation of lower yield stress, σ_{LY} with grain diameter, d, in Fe, Mo, and Cb has been found experimentally by many investigators to obey the Petch equation

$$\sigma_{LY} = \sigma_o + k_Y d^{1/2}$$

where σ_o is the stress resisting glide of unlocked dislocations and k_Y is thought to be a measure of the stress required to unlock dislocations from their Cottrell atmosphere. For Fe, Mo, and Cb, the tendency towards mechanical twinning and brittle fracture is higher for metals having high values of k_Y than for metals having relatively low values of k_Y. Since Ta is known to be exceptionally ductile and reluctant towards mechanical twinning, values of k_Y for high-purity Ta at 298°, 203°, and 77°K have been determined by measurements of the yield stress of specimens with a wide range of grain sizes.

A strong dependence of twinning on grain size at 77°K has been found. Profuse twinning occurs in coarse-grain specimens even at low rates of deformation, but twinning is absent in fine-grain specimens even at high strain rates. The failure to induce twinning in fine-grain specimens by high strain rates at 77°K cannot be attributed to the temperature rise in the specimen during rapid loading.

Values of k_Y determined from the variation of lower yield stress with grain diameter are: 2.3 x 10^7 dynes per $cm^{3/2}$ at 298°K, 3.8 x 10^7 dynes per $cm^{3/2}$ at 203°K, and 5.2 x 10^7 dynes per $cm^{3/2}$ at 77°K. The effective surface energy, γ, for cleavage crack propagation in tantalum has been estimated to be $> 2.5 \times 10^6$ erg per sq cm. Comparisons of the values of k_Y and γ for tantalum with those determined for Fe, Mo, and Cb show that k_Y for tantalum is substantially lower than might be expected and that γ is two orders of magnitude higher than those for other bcc metals. Therefore, it is reasonable to attribute the high ductility of tantalum (with 90 pct reduction in area at 77°K) to the large effective surface energy for crack propagation rather than the low value of k_Y assumed previously.

Micro-Strain Behavior of Aluminum: *R. C. Ku* and *Norman Brown*, University of Pennsylvania.

The deformation of aluminum single crystals and polycrystals was investigated in the region of microyielding. The lowest resolved shear stress at which dislocation movement could be detected in single crystals was about 60 psi and the lowest tensile stress for polycrystals was about 400 psi. This lowest stress varied slightly in single crystals which received prior strains ranging from 10^{-4} to 10^{-2} and was independent of strain for the polycrystals. Temperature had no effect on the stress to just move dislocations in polycrystalline specimens. The stress at which a permanent strain of 10^{-6} could be detected was strongly temperature and strain dependent.

The lowest stress at which dislocation movement can be detected is associated with the average frictional stress to move dislocations on the order of several to a thousand atomic distances. The frictional stress probably arises from impurities and the basic resistance of the lattice to the movement of dislocations. The stress at which a permanent strain is first detected is associated with the generation of dislocations and the interaction of the moving dislocations with the forest dislocations.

WEDNESDAY, OCTOBER 25

Deformation of Tantalum Single Crystals: *R. M. Rose* and *J. Wulff*, Massachusetts Institute of Technology, and *D. P. Ferris*, E. I. du Pont deNemours and Co.

Single crystals of tantalum were grown by multipass zone melting in an electron beam apparatus. The total interstitial impurity level was 0.003 to 0.007 wt pct, and the total substitutional elements were less than 0.001 wt pct except for less than 0.1 wt pct W.

Tension tests at 4.2×10^{-4} per second in the temperature range 77° to 300°K resulted in a resolved lower yield stress of 7300 psi at 300°K and 45,400 psi at 77°K. All crystals fractured with a chisel edge after uniform deformation which increased from 1 or 2 pct at 77°K to over 20 pct at 300°K. This and slip traces indicate that deformation is more inhomogeneous at the lower temperature.

Deformation occurs exclusively by slip on {110} planes in a <111> direction regardless of crystallographic orientation. Cross slip is frequent, particularly for those orientations where the resolved shear stress on the cross system is comparable with that on the primary plane. Double cross slip (composite slip) in the preyield strain region is capable of circumventing interstitially locked dislocations if the above condition for composite slip is favorable. This mechanism explains the orientation dependence of the existence and magnitude of an upper yield point, which the experimental data reveal and which is not predicted by the existing theory of interstitial locking. Also not predicted by theory is the observation that prestraining at 300°K does not remove the upper yield point in subsequent tension at 77°K. In fact, prestraining is even capable of causing a yield point at a lower test temperature. This behavior can be rationalized in terms of the inhomogeneous nature of low temperature deformation and the exhaustion of mobile dislocations during prestraining.

Since it is very likely influenced by thermally activated cross slip, the amount of composite slip may be associated with the steep rise of yield stress and the greater effectiveness of interstitial locking in bcc metals as temperature decreases.

Yielding and Plastic Flow in Single Crystals of Tungsten: *R. M. Rose* and *J. Wulff*, Massachusetts Institute of Technology, and *D. P. Ferris*, E. I. du Pont de Nemours and Co.

The effects of orientation on the stresses, activation energies, and activation volumes for yielding and plastic flow of tungsten single crystals were investigated. Tensile tests showed the proportional limit to be a function of orientation: crystals whose tensile axes were near the <110> direction were roughly three times as strong as <100> crystals. The <110> crystals did not strain harden, and displayed yield phenomena, whereas the other orientations yielded smoothly and showed considerable strain hardening. The fracture mode was cleavage, except for crystals near the <110>, which gave *chisel* fractures. The activation energy for yielding was approximately one-half an electron volt at all orientations, and the volume of activation was $(12\text{-}14)b^3$, except near the <110>, where it was $7b^3$. Activation volumes increased after yielding. The activation energy for plastic flow at approximately 5 pct plastic strain varied with orientation, and was highest (2.4 ev) at the <100> orientation. All activation energies were found to be dependent on both stress and temperature. The available theoretical models for yielding and strain hardening of body-centered cubic metals were found to be insufficient to the interpretation of the observations herein. A new explanation is proposed, based on the dislocation reaction

$$\frac{a}{2}[111] + \frac{a}{2}[\bar{1}\bar{1}\bar{1}] \rightarrow a[100]$$

The authors gratefully acknowledge the sponsorship and financial support of the Office of Ordnance Research, and the National Science Foundation Cooperative Fellowship which made it possible for R. M. Rose to engage in this investigation.

Deformation Twinning and Stacking Faults in Body-Centered-Cubic Transition Metals and Alloys: *E. E. Underwood*, Battelle Memorial Institute.

Nine transition metals and fifteen alloys based on Mo, W, and Cr were investigated for their twinning characteristics. A conical hardness impression was used to supply the compressive strain to induce the formation of deformation twins.

An analysis of the strains around the hardness impression gave the equation: ϵ (pct) = $19.2/R^3$, where R is the normalized distance from the center of the hole. A stereographic analysis of the twin traces appearing in a large crystal showed that the traces coincide with {112} planes. A quantitative metallographic procedure was devised to measure the amount of twinning as a function of distance from the hole. Twin density is expressed in terms of length of twin traces per unit area.

Slopes of the hardness vs composition curves (called the *hardening coefficients*) were plotted against the atomic number of the respective solutes. A minimum in hardening coefficient was found to occur for all alloys near W, and a maximum near, or after, Pt. Hardening coefficients vary in the same order as the *extent* of twinning, i.e., the greater the hardening capability of the alloy, the greater the distance from the hole that twinning is observed. The actual hardnesses of the alloys decrease in the order of the base metals, W, Mo, and Cr, as do the *intensity* of twinning.

The amount of twinning measured around holes of various diameters varies widely; however, when replotted to a normalized distance scale (in multiples of the particular radius), the curves coincide. This result is expected on the basis of the derived strain vs distance curve. The critical plot of amount of twinning vs compressive strain shows twinning occurring at small strains, increasing rapidly, then leveling off at higher strains. According to the strain vs distance analysis, there should be a small, finite strain necessary for twins to occur.

This work was supported by the Battelle Memorial Institute.

Yielding and Flow of Sapphire ($\alpha - Al_2O_3$) Crystals: *Hans Conrad*, Atomics International.

The plastic flow of sapphire crystals was investigated in tension at 1100° to 1500°C and at strain rates of 5×10^{-6} to 5×10^{-2} sec^{-1}. The stress-strain curves exhibited a proportional limit, followed by discontinuous yielding and subsequent flow with strain hardening. The behavior at all strains could be described by an equation of the form

$$\dot{\gamma} = \frac{A}{T}(\tau - h_\gamma)^n e^{\frac{-120{,}000}{RT}}$$

where $\dot{\gamma}$ is the strain rate, τ is the applied shear stress, h_γ is the increase in stress due to strain hardening, and A and n are constants. n was approximately 4.0 for the proportional limit and the upper yield stress and 4.5 for the lower stress and subsequent flow. The form of this equation and the values of the constants supports Weertman's dislocation climb mechanism as the rate-controlling mechanism for yielding as well as subsequent flow.

This work was supported by the U.S. Atomic Energy Commission.

Kinking and the Fracture of Ionic Solids: *R. J. Stokes* and *C. H. Li*, Minneapolis Honeywell Regulator Co., and *T. L. Johnston*, Ford Motor Co.

A single crystal undergoing plastic bending develops constraints due to the gradient in lateral contraction across the beam. These constraints result in lateral stresses which may be relieved by the process of *anticlastic kinking*. Anticlastic kink boundaries consisting of <110> {121} edge dislocations form along the corners of rock salt ionic single crystals by the interaction of two systems of <110> {110} glide dislocations moving on intersecting planes. One system is responsible for slip in the main part of the crystal, the other is confined to its corner.

Temperature affects the structure of the kink boundaries and their subsequent role in initiating fracture.

1) At high temperatures (~0.3 T_m). The resultant edge dislocations in the boundary can move over their {121} slip planes and the kinks become sharp. The resultant dislocations are ineffective barriers to slip and the crystals are ductile.

2) At low temperatures (~0.1 to 0.2 T_m). The resultant edge dislocations are immobile and the kinks consist of a diffuse array. The resultant dislocations provide strong barriers to slip and cracks nucleate at the kink boundary.

3) At very low temperatures (~0.1 T_m). Fracture occurs before the second set of {110} <110> glide dislocations have been activated to generate anticlastic kinks. Relaxation of the lateral stress results in a complex fracture.

Compatible observations of the change in fracture behavior under bending over different temperature ranges have been made on NaCl, LiF, MgO, and AgCl.

This work is supported by O.N.R.

WEDNESDAY, OCTOBER 25

Crack Nucleation in Magnesium Oxide Bi-Crystals: *T. L. Johnston*, Ford Motor Co., and *R. J. Stokes* and *C. H. Li*, Honeywell Regulator Co.

Magnesium oxide bi-crystals having a wide range of misorientation were tested under three-point loading. They were heat treated and handled carefully so that the only fresh, and therefore mobile, dislocations were those introduced along the line of the loading beams. The interaction of slip bands coming from the center loading beam with the grain boundary were studied on the compression surface.

When a single slip band impinged upon the boundary one of three things happened depending upon the misorientation.

1) For small misorientations (i.e., a simple tilt boundary). The shear stress concentration ahead of the pile-up nucleated dislocation loops and thereby slip bands in the adjacent grain. This relaxed the stress concentration and cracks did not form.

2) For medium misorientations (i.e., a twist plus a 10° to 15° tilt boundary). The tensile stress concentration beneath an edge slip band nucleated a *transgranular* crack in the adjacent grain. Under compression, this cleavage crack decelerated and generated dislocation loops which developed into slip bands in the adjacent grain.

3) For large misorientation (i.e., a large twist plus a 20° to 30° tilt). The tensile stress concentration beneath an edge slip band nucleated an *intergranular* crack.

In particular this work has shown that a crack can nucleate at a grain boundary in MgO at room temperature by the pile-up of a *single* band of edge dislocations according to classical Zener model.

Oxygen Precipitation Effects on the Deformation of 'Dislocation-Free' Silicon: *J. R. Patel*, Bell Telephone Laboratories, Inc., and *A. R. Chaudhuri*, Westinghouse Electric Corp.

Silicon crystals grown from quartz crucibles usually contain about 10^{18} per cu cm of oxygen. When specimens from such crystals are heat treated for prolonged periods at 1000°C, there is evidence to show that oxygen precipitates to form a quartz-like structure. Precipitation of oxygen has a pronounced effect on the tensile deformation characteristics of silicon. The large yield drop that is observed in normal as-grown silicon at a temperature of say 800°C is diminished by successive amounts depending on the time of annealing at 1000°C. Since the measured velocities of dislocations are not altered by heat treatment, the effect of precipitation can be to either 1) increase the initial dislocation density of the crystals, or 2) to provide very effective nucleation sites for the generation of dislocations during deformation. In dislocation-free crystals of silicon, the effect of precipitation is to increase the initial dislocation density depending on the heat treatment time. For crystals that contain a few dislocations, the effect of oxygen precipitation is not to increase the initial dislocation density, but rather to provide active sites for the generation of dislocations during deformation. The precipitates may be dissolved by heat treatment at 1300°C. The effect of redissolving precipitates on the deformation characteristics will be discussed. Part of this work was performed at the Raytheon Research Division, Waltham, Massachusetts.

THURSDAY, OCTOBER 26

Deformation II

9:00 am to 12:00 m — **Terrace Room**

Chairman:
A. McEvily, Ford Motor Co.

Mechanical Properties of NaCl-AgCl Alloys: *R. J. Stokes* and *C. H. Li*, Honeywell Regulator Co.

Both NaCl and AgCl possess the rock salt structure with similar lattice parameters and are reported to form a single solid solution over their whole composition range. Single crystals of the pure salts show markedly different mechanical behavior at room temperature. NaCl is ductile only when the surface is flaw-free and deforms by straight slip. AgCl is always ductile and deforms by straight slip. We have studied the mechanical properties of their alloys.

NaCl-AgCl alloy single crystals were grown by the Stockbarger technique. Small parallelipiped specimens prepared by cleavage were tested in compression. It was found that the yield strength versus composition followed an unexpected bell-shaped curve. The yield strength increased at a normal solid solution strengthening rate for small additions of NaCl and AgCl but in the 15 pct NaCl and 15 pct AgCl regions it increased rapidly to a very high maximum of 25,000 psi which was maintained between 25 to 75 pct NaCl. This represented a 60-fold increase in strength over the end components. It will be shown that this high strength is due to *precipitation hardening*.

Single crystals between 15 and 85 pct NaCl composition have a milky white appearance when grown which clears following a solution heat treatment and returns on standing at room temperature. The precipitation reaction has been studied by X-ray and microscopic techniques. The phase diagram for NaCl-AgCl is not a simple solution but contains a two phase field.

The change in slip mode from wavy to straight slip has been found to occur at 50 pct NaCl.

This work is supported by O.N.R.

Strength and Creep Behavior of Silver-Alumina Composites Above the Melting Point of Silver: *H. R. Peiffer*, RIAS

Dispersed phase materials have been recognized as having good high temperature mechanical properties. Ideally, one would like to have these materials strong enough to be used at temperatures very near to the melting point of the continuous phase metal. Certain silver-alumina composites show mechanical strength up to and beyond 960°C, the melting point of silver, which is the continuous phase in these materials.

The ultimate tensile strength of alloys containing 15 to 20 wt pct *Linde B* alumina have been studied above 960°C. Shoulder grip specimens were gripped by means of wires and pulled to fracture in air at temperatures in excess of 960°C. The average strength of these materials was constant with temperature. The strengths were 3.8×10^8 and 0.4×10^8 dynes per sq cm for the materials containing 20 and 15 wt pct alumina respectively.

The creep behavior of the 20 wt pct alumina material was also studied by allowing a rectangular bar to deform in bending under its own weight and under additional loads. The materials always

THURSDAY, OCTOBER 26

deformed for a given length of time and then appeared to cease deforming. The initial creep rate, $\dot{\epsilon}$, can be expressed by

$$\dot{\epsilon} a \sigma^2 e^{-E/kT}$$

where σ is the applied stress and E the activation energy for the process. The activation energy is of the order of 1.7 to 2.1 ev. Interestingly enough the creep behavior is very similar to that of solid metals.

A third interesting phenomenon was observed upon cooling these materials through the melting point of silver. Single crystals of the silver matrix appear to grow relatively easily even without much care taken to prepare a suitable temperature gradient. Attempts are being made now to study slip line formations in these crystals.

Some Observations of Yield Points in Aluminum at High Temperatures: *R. H. Bush*, Ford Motor Co., and *R. A. Huggins*, Stanford University.

Pronounced yield points have been observed for single crystals of high purity aluminum tested in tension at temperatures between 450° and 650°C. The magnitude of the yield point was found to depend upon the test temperature, the extension rate, the amount and type of prior strain, and, indirectly, on the sample orientation. The yield point behavior is found to change with increasing strain. The observed phenomena will be discussed in terms of existing theories of yield point mechanisms.

Lüders Band Propagation in Low Carbon Steels: *J. F. Butler*, Jones & Laughlin Steel Corp.

Lüders band propagation during the yielding of low carbon steels has been studied through the tensile straining of wire and thin sheet specimens. The progress of Lüders front movement in wires and the orientation of simple Lüders fronts on sheet surfaces were recorded at high strain rates by photography and at low strain rates by visual observation. Contrast between the yielded and unyielded portions of the specimen was obtained through the use of a brittle lacquer which cracked upon specimen deformation. Although Lüders fronts always were propagated from each grip in the specimen of uniform gage, Lüders fronts could be artificially nucleated by bending prior to tensile deformation.

It was shown that by increasing the number of Lüders fronts while keeping the other variables constant, the stress to propagate Lüders fronts (the lower yield stress) decreased. Therefore, in large grained specimens where many diffuse Lüders fronts form, the lower yield stress cannot properly be compared to the stress to propagate two Lüders fronts in small grained samples.

Specimen inhomogeneities in grain size (d) and lattice frictional stress on dislocations (σ_o) were deliberately introduced along wire specimens in order to determine their effect upon Lüders front propagation and the stress-strain relationships during yielding. Homogeneous specimens also were used to determine the relationships between the stress to propagate Lüders fronts (σ_L), their velocity (v_L), the strain rate ($\dot{\epsilon}$), gage length (l_o) and yield point elongation (ϵ_L) for several steels with respect to the variables σ_o and d of the Petch equation:

$$\sigma_L = \sigma_o + kd^{-1/2}$$

Some Observations on Complex Damping Effects in Fe-Mn-N Alloys: *J. F. Enrietto*, Jones & Laughlin Steel Corp.

It is well known that the presence of small amounts of substitutional alloying elements can profoundly affect the profile of the internal friction spectrum in the vicinity of the Snoeck peak in bcc metals. The observed broadening and shifting of the Snoeck peak have been explained as being the result of two or more internal friction maxima occurring near enough to each other so that they overlap. In general these explanations inherently contain two fundamental assumptions: 1) each process which gives rise to a subsidiary peak takes place with a single relaxation time, and 2) there are only two distinct types of lattice sites involved.

We have carried out some experiments to determine the validity of these assumptions in the Fe-Mn-N system. Alloys containing 0.15 to 0.75 pct Mn and 0.002 to 0.035 pct N were prepared and their internal friction at frequencies of 0.5 to 2 cps measured by means of a conventional torsional pendulum. It was impossible to fit a given set of theoretical conditions to all of the experimental curves and still retain the concept of single relaxation times for all the processes involved. Furthermore, the peak profile changed with increasing nitrogen concentration. It can be shown this would not be expected if nitrogen was distributed only between two types of sites in the ferrite lattice.

The results of these experiments thus show that the two assumptions mentioned previously are perhaps unwarranted in the sense that they impose too rigorous a constraint on the damping mechanisms. It is suggested that the concept of single relaxation times for each individual process be abandoned along with that of only two sites. By treating each Mn atom as a center of inhomogeneity (which can extend several atomic distances) the experimental results can be more fully explained.

Dislocation Damping in Deformed Low Carbon Steel: *W. J. Bratina* and *D. Mills*, Ontario Research Foundation.

A study was made of the effect of plastic deformation on dislocation damping in low carbon steel (SAE 1020) employing ultrasonic techniques in the low megacycle frequency range. In general, it was necessary to introduce plastic deformation in this material before the damping attributed to dislocations could be detected. It was certainly absent or negligible in well annealed or in well strain-aged material. Although the simultaneous presence of damping associated with mechanisms of magnetic nature complicates the study of dislocation damping, the information obtained by ultrasonic techniques seems to be difficult to obtain by any other method.

Of main interest was the elastic strain induced dislocation damping; this was studied at various time intervals after plastic deformation, i.e. in incompletely strain-aged material. In this respect the change of damping with time (decay) was studied for various conditions. Further, a rise of dislocation damping versus number of load repetitions was investigated. All these experiments were performed at room temperature. The effect of moderately increased temperatures and the effects of the degree of plastic deformation on the dislocation damping were also investigated.

All the results obtained seem to be qualitatively explained by a dislocation loop-pinning point (interstitial and/or precipitate) interaction. The experimental results will be discussed in detail with reference to this interaction.

This work supported by the Dept. of Planning and Development of the Province of Ontario, Canada.

Metallographic Studies of the Fatigue Process in Copper: *H. M. Bendler*, General Motors Corp., and *W. A. Wood*, University of Melbourne

The surfaces of both polycrystalline and single crystal cylindrical copper fatigue specimens have been observed by optical and electron metallography at various stages of the fatigue life of the specimens. The polycrystalline samples were prepared from OFHC copper of 0.25-in. diam machined to give a central test portion 1.25-in long and 0.2187 in. diam. After annealing in hydrogen and electropolishing in phosphoric acid the specimens were subjected to alternating torsion. Specimens which had been cycled at different amplitudes and for different fractions of their fatigue life were examined for surface deformation (slip bands, fissures, and fatigue cracks) using two stage carbon replicas. By silver plating a specimen at the end of a run and then grinding and polishing a narrow (0.01 in. wide) flat on the surface, the slip bands and fissures could be studied in cross section. The relation of surface deformation to sub-surface structural change may be revealed in this manner.

If a superimposed tensile stress is applied to the polycrystalline specimen while it is being fatigued, the development of the fissures is much enhanced and the fatigue life markedly shortened. Under these conditions examples have been noted of cracks opening entirely beneath the surface.

Oriented copper single crystals 3 in. long and 0.375-in. diam were grown from the melt and provided with a 1 in. gauge length by electrolytic machining. Crystals of both [100] and [111] axial orientation were cycled in torsion and replicas made of the surface for both optical and electron metallographic examination. While the disposition of the slip bands on the surface showed that, in general, the resolved shear stress law was obeyed by the slip produced by fatigue, localized interactions produced slip on secondary systems that was apparently associated with the formation of fatigue cracks.

Effect of Environment on the Fatigue of Lead and Aluminum: *K. U. Snowden*, Franklin Institute Laboratories.

Results will be given of experiments on the influence of gaseous environments at normal and reduced pressures on the fatigue life and deformation of high purity polycrystalline lead and aluminium.

The air-pressure dependence of the fatigue life of aluminium showed a steady increase in life with decrease of air pressure which is in agreement with the dependence found for aluminium and copper by other workers. In contrast, the dependence for lead showed three distinct ranges: 1) a high-pressure range in which the life is constant (atmospheric pressure to approximately 0.15 mm Hg); 2) an intermediate range in which the life in-

THURSDAY, OCTOBER 26

creases rapidly with decreasing pressure (approximately 0.15 to 5 x 10^{-5} mm Hg); and 3) a low-pressure range in which the life is unaffected by further reduction of pressure (approximately 5 x 10^{-5} to 2 x 10^{-6} mm Hg.).

The stepped form of the curves for lead is thought to be associated with specific changes in the nature of the fatigue deformation produced by variation in the rate of reaction between certain adsorbed oxygen molecules and the metal at different gas pressures.

Metallographic observations were made of the changes in slip distribution, grain-boundary phenomena, and development of cracking which accompanied changes of gas pressure.

Effect of Hydrogen on the Creep Properties of Nickel-Base Materials at Elevated Temperatures: *H. E. McCoy* and *J. R. Weir*, Oak Ridge National Laboratory.

The proposed use of hydrogen as a reactor coolant makes it imperative that the mechanical behavior of structural materials in hydrogen environments at elevated temperatures be known. This paper is concerned with the properties of nickel-base materials, primarily *A* nickel and Inconel. The characteristic room-temperature hydrogen embrittlement which occurs in many of these alloys is discussed briefly and several possible mechanisms whereby hydrogen may influence the mechanical behavior at elevated temperatures are postulated.

Data are presented which show that the high-temperature creep strength of nickel and Inconel is less in a hydrogen environment than in an argon environment. This decrease in strength is manifested through both an increase in the secondary creep rate and a decrease in the rupture life. No significant effect on the ductility is observed. The magnitude of this effect appears to be dependent upon the rate of deformation, with larger effects being observed at lower strain rates. These results are reviewed in light of the proposed mechanisms.

Elevated Temperature Tensile and Creep-Rupture Properties of the FS-82 Columbium Alloy: *R. L. Jones*, General Dynamics.

The base metal and fusion weldment tensile properties of the FS-82 columbium alloy (Cb + 33Ta + 0.7Zr) have been determined from ambient temperature to 2600°F. Base metal tensile properties were determined using two different heat-up periods prior to test: 30 sec and 30 min. In general, the tensile properties determined using the short heat-up period were considerably lower than those found using the long heat-up period at temperatures below 1800°F but much higher at higher temperatures. This behavior was related to strain aging at temperatures below 1800°F and recovery and/or recrystallization at temperatures above 1800°F. Creep-rupture properties of the base metal and fusion weldments were determined at 1800°, 2000°, and 2200°F. Extrapolation techniques were employed to extend the data to cover temperatures, times and stresses not investigated in the experimental program. The creep behavior of the base metal was analyzed and the activation energy for creep was found to be 121,-300 cal per mole. This was found to be in excellent agreement with the activation energy for self diffusion of columbium (105,000 ± 3000 cal per mole) and/or the activation energy for self diffusion of tantalum (110,000 cal per mole). These data strongly suggest that creep of the FS-82 columbium alloy is diffusion controlled above about 1800°F (0.45 T_m).

Electron beam fusion welds of FS-82 were found very ductile and free from contamination. Tungsten-arc gas shielded fusion welds (TIG Welds) were contaminated slightly by gas pick up during welding but retained considerable ductility. Tensile weld efficiency of the TIG weldments decreased from 94 pct at ambient temperature to about 75 pct at 1800°F. Electron beam weld efficiencies were considerably lower; decreasing from 64 pct at ambient temperature to 54 pct at 1800°F. The rupture life of the fusion welds appear to be higher than the base metal rupture life at temperatures over 2000°F, but appear to be lower at lower temperatures. However, more data are required before this conclusion can be considered valid.

Internal Friction Analysis of Residual Stress in Alloys: *I. G. Hendrickson*, The Boeing Co.

Internal friction measurements, as made utilizing the unrestrained—fundamental—torsional mode of vibration and employing a *tuned Q* technique, have proved very sensitive to lattice distortion and stresses in metal alloys produced by various heat treatments and also by introduction of small amounts of hydrogen and other interstitials into the metal alloys. A range of *relative Q* values from 13,800 to 24,100 was obtained for a series of specimens of 4330 steel with a Rockwell C hardness range of 50-41 (respectively). The electrolytic charging of 4330 steel R_c 49, with a 4 pct H_2SO_4, saturated P_2S_5 solution under a 3.0 v potential for 2 min produced a decrease in relative Q from 14015 to 13140 and for a total of 5 min to 12486. A recovery of relative Q from 12486 to 12689 was observed over a 10 min period following the 5 min charge and is believed to be due to the diffusion or escape of hydrogen from the charged specimen. Materials under investigation in this study include 4330 steel, 1020 vacuum melt, and Ferrovac E.

Phase Transformation and Pressure Effects

9:00 am to 12:00 m — **Ballroom**

(Based on Submission of Research Abstracts)

Chairmen:

G. R. Speich, United States Steel Corp., and *J. W. Spretnak*, Ohio State University.

Shock Wave-Derived T-P Phase Diagram of Iron: *P. C. Johnson, B. A. Stein*, and *R. S. Davis*, Arthur D. Little, Inc.

Shock waves were used to investigate the T-P phase diagram in Armco iron. The technique used involved metallographic investigations of specimens shocked at temperatures between 78° and 1158°K. An apparent triple point was found at about 500°K and 115 kilobars. This is substantiated by metallographic evidence.

Calculation of the Effects of Hydrostatic Pressure on Solid Phase Transformations in Thallium and Tin: *Larry Kaufman*, Manufacturing Laboratories, Inc.

At room temperature and one atmosphere, Tl has the ϵ (hcp) crystal structure. Lumsden's data on the free energy difference between the α (fcc) and the ϵ (hcp) forms of Tl in combination with volumetric data from X-ray measurements on ϵ Tl,αPbTl, and α BiTl alloys, and unstable α Tl were used to calculate the transformation pressure at 300°K. These data suggest that at 300°K, $\Delta F_{Tl\alpha\rightarrow\epsilon}$ is 24 cal per mol and $\Delta V_{Tl\alpha\rightarrow\epsilon}$ is -0.026 cu cm per mol, indicating a transition from ϵ to α at 38 kilobars and 300°K. Kennedy and LaMori have recently redetermined the transition pressure for Tl, first reported by Bridgman, at 298°K and 36.7 kilobars. These considerations indicate that the high pressure form of Tl has the α (fcc) structure, however the identity of the high pressure form has not yet been established. On the basis of Lumsden's free energy data the stacking fault energy of Tl should be small, and temperature dependent.

Calculations have been carried out for the transformation of tetragonal tin, τ Sn, to α (fcc) Sn at high pressures as follows: The one atmosphere form, τ, melts at 505°K where the heat of fusion is 1720 cal per mol. At 505°K the molar volume of τ Sn is 16.6 cu cm per mol and the volume change on fusion, $\tau\rightarrow$L, is 0.465 cu cm per mol. Geguzin and Pines estimate the melting temperature of α Sn at one atmosphere as 350°K. Taking the entropy of melting of α Sn as 2.20 cal per mol°K, the entropy change for the $\alpha\rightarrow\tau$ (fcc$\rightarrow$tetragonal) reaction is 1.21 cal per mol°K. Kubachewski has shown that for close packed metals ($\Delta S \cdot v/\Delta V$) = 60 cal per mol°K where ΔS and ΔV are the entropy and volume change of fusion and V is the molar volume of the solid at the melting point. This information has been used to compute (dT/dP) for the $\tau\rightarrow$L, $\tau\rightarrow\alpha$, and $\alpha\rightarrow$L reactions as well as the τ-α-L triple point. The results are + 3.3, − 2.8, and + 6.6°K per kilobar respectively with a triple point at 46 kilobars and 655°K.

Recently Dudley and Hall, and Kennedy and Newton have found a triple point between τ Sn, Sn II, and liquid Sn. Kennedy and Newton measured dT/dP for the $\tau\rightarrow$L, $\tau\rightarrow$II, and II$\rightarrow$L reactions and determined the triple point. Then results are + 2.7, − 3.0 and + 6.0°K per kilobar respectively with the triple point near 31 kilobars and 580°K. The calculations indicate that SnII is α (fcc) Sn. Direct determination of the crystal structures of SnII must await the development of high-pressure-high temperature X-ray techniques.

This work has been sponsored by the Metallurgy Branch, ONR Washington, D. C. under contract Nonr 2600(00) and by Materials Central, WADD, Dayton, Ohio under contract AF33(616)-6837.

THURSDAY, OCTOBER 26

Effect of High Pressure on the Martensitic Transformation in Iron-Carbon Alloys: *S. V. Radcliffe* and *M. Schatz*, Manufacturing Laboratories, Inc.

The effect of a hydrostatic pressure of 42,000 atm on the kinetics of the athermal transformation of austenite to martensite have been investigated for high-purity iron-carbon alloys containing 0.3 to 1.23 wt pct of carbon. At this pressure, depending on the composition, the martensite start temperature (M_s) is depressed 150° to 200°C below that at atmospheric pressure. The measured depression of M_s is in agreement with that calculated from a consideration of the effect of pressure on the thermodynamics of the martensitic transformation.

At high pressure, in contrast with the behavior at atmospheric pressure, the extent of the transformation as a function of the temperature of cooling below M_s is dependent on composition. In addition, there is some evidence that high pressure reduces the amount of transformation attainable in the alloys of higher carbon content.

This work was sponsored by the Metals and Ceramics Laboratory, Materials Central, Wright Air Development Division, ARDC, U.S. Air Force.

Occurrence of Pseudo-Eutectoid Reactions Above the Eutectoid Temperatures in Ternary Iron-Carbon-Base Alloys: *H. Warlimont*, United States Steel Corp.

In hypo-eutectoid or hyper-eutectoid ternary alloys of iron and carbon the formation of the pro-eutectoid phase, ferrite or cementite, above the eutectoid temperature can give rise to a reaction by which the alternate phase, cementite or ferrite, also forms. The resulting microstructures are somewhat similar to pearlite in appearance. They consist of a matrix of the equilibrium phase which contain *occlusions* of the non-equilibrium phase, partially in a lamellar arrangement, and are sometimes called *occlusive* or *porous* constituents.

Electron microscopy of surface replicas and thin films, selected area diffraction, and electron micro-probe analyses have yielded detailed information about changes in the austenite during transformation, and about the structural features of the reaction products.

Partitioning of substitutional alloy components and the rate of their redistribution has been found to be the main reason for the shifts of equilibria at the phase-interfaces which result in the formation of *occlusive* constituents. By diffraction of thin-film specimens, crystallographic relationships between the phases in the product have been observed. They are interpreted to be the result of interface energy minimization when the non-equilibrium phases are nucleating.

The occurrence of this pseudo-eutectoid reaction can be predicted if the equilibrium relationships and diffusivities of the components are known. Reactions of this type sometimes give rise to anomalous microstructures in technical applications.

Thermodynamic Interpretation of the Allotropic Transformation of Zirconium-Rare Earth Alloy Systems: *D. J. Lam*, Argonne National Laboratory, and *A. A. Burr*, Rensselaer Polytechnic Institute.

The effects of solute elements on the allotropic transformation temperature has been studied qualitatively in terms of the thermodynamic nature of the systems involved. In this investigation, the problem of evaluating the relative stability of the alpha and beta phases of zirconium on alloying with rare-earth elements is similar to that of making a comparison of the effects of a solute on two different solvents, except that the solubilities in the allotropes are competitive.

An examination of the zirconium-rich portion of the binary phase diagrams with the solutes yttrium, neodymium, dysprosium, holmium, and erbium has been made. Alpha and beta phase boundaries for these alloys have been used to estimate values of Q_B, the difference of the differential heat of mixing of an alloying element in the allotropic forms of the solvent metal. Periodic variations of Q_B values with atomic number of the solutes have been examined, and the theoretical phase boundaries were calculated using the experimental Q_B data. The results of the theoretical and experimental phase boundaries will be compared and discussed.

This abstract is based on work performed at Rensselaer Polytechnic Institute sponsored by the Atomic Energy Commission under contract AT(30-1)-2159.

Theory for the Allotropic Transformation of Titanium: *R. H. Willens*, California Institute of Technology.

A vacuum high-temperature X-ray diffractometer has been designed and constructed to study metals, sensitive to oxygen and nitrogen contamination, up to 1200°C. An investigation of titanium to 650°C has shown that between 0° and 400°C, the mean coefficients of thermal expansion perpendicular and parallel to the c axis are 9.41 x 10^{-6} per °C and 11.18 x 10^{-6} per °C, respectively and for a random polycrystalline sample, the mean linear expansion coefficient is 10.0 x 10^{-6} per °C. A study of the variation of the c/a ratio with temperature reveals that this ratio increases linearly with temperaure up to 400°C. Above 400°C the c/a ratio rapidly increases with temperature. This variation of the c/a ratio is correlated with other magnetic and electrical properties of titanium to formulate a band model. This band model assumes that the electrons at the top of the band at 0°K are on overlapping Fermi surfaces and there is a small number of unfilled states available to the electrons below an energy gap associated with the c direction in $\vec{k}$-space. On the basis of this model it is shown that the electrons can give an appreciable positive contribution to the free energy as the transformation temperature is approached and it is proposed that this may be the main factor causing instability of low temperature modification of titanium. Although the analysis is confined to titanium, the ideas formulated about allotropic transformations can probably be extended to other metals such as zirconium and hafnium.

Electron Metallographic Examination of Ti–13V–11Cr–3Al: *L. E. Tanner*, Manufacturing Laboratories, Inc., and *J. E. Coyne, Jr.*, Wyman-Gordon Co.

In recent studies of the isothermal decomposition of the β phase during aging of the titanium-base alloy B120 VCA (12V-11Cr-3Al) indications of the presence of the ω transition reaction were reported. However, the evidence was not sufficiently definitive for positive identification due to the limited resolution of light metallographic techniques and difficulties with X-ray diffraction determinations. In the current work electron microscopy was employed to study microstructures developed at aging temperatures between 700° and 900°F. The reaction sequence in this temperature range is believed to be:

$\beta \rightarrow \beta + \omega \rightarrow \beta + \omega + \alpha \rightarrow \beta + \alpha \rightarrow \beta + \alpha + TiCr_2$

A structure was observed in the early stages of aging that was distinguishable from the well known α and Ti Cr_2 decomposition products. This structure could not be classified as a precipitate but appeared to be a surface disturbance of the β phase. The ω transition phase is known to be coherent with the matrix β and therefore the above effect is believed to be the result of the coherency strains developed at the β-ω interfaces.

Electron Transmission Studies of Fe–Cr–Ni Alloys: *John Dash* and *H. M. Otte*, RIAS.

Partially transformed high purity Fe-Cr-Ni alloys have been examined by electron microscopy and selected area diffraction of suitably thinned samples. Results obtained from a 15/10, an 18/12, and a 5/20 alloy show that the dislocation density in the martensite is exceedingly high. In the 18/12 alloy, a few dislocations may be seen in the adjoining austenite which, before transformation, contained essentially none. No twins or twin faults were found in the martensite crystals, and adjacent crystals were found to have the same orientations. Diffraction patterns from the austenite (A), martensite (M), and hcp epsilon phase were obtained. The following approximate orientation relationships were observed for the 18/12 between the 3 structures:

$(110)_A$	$\|\|$ $(111)_M$	$\|\|$ $(12.0)_\epsilon$
$[1\bar{1}2]_A$	$\|\|$ $[11\bar{2}]_M$	$\|\|$ $[\bar{1}0.0]_\epsilon$
$[\bar{1}11]_A$	$\|\|$ $[110]_M$	$\|\|$ $[00.1]_\epsilon$

In the 18/12 the bcc martensite was generally found associated with the hcp structure, which formed in long bands parallel to the $(111)_A$ plane. The bcc appeared to form across these and clearly did not have a $(111)_A$ habit plane. Though adjacent crystals of bcc martensite often showed different contrast in the electron microscope, their orientation was found to be the same. This effect was attributed to a difference in the dislocation configuration.

This research was supported in part by the A.E.C. under Contract No. AT(30-1)-2531.

THURSDAY, OCTOBER 26

Delay Time in the Alpha to Gamma Transformation in Iron: *O. A. Boedtker* and *Pol Duwez*, California Institute of Technology.

Iron, containing 66 ppm of carbon and 190 ppm of nitrogen is heated from an initial temperature in the alpha phase into a final temperature within the gamma phase. The rise time is about 700 microseconds. The allotropic transformation which is detected by a sudden heat release, occurs a certain time after the final temperature is reached. The relationship between this delay time and the final temperature follows an Arrhenius type rate equation with a heat of activation of 20,500 ± 1400 cal per mole. The effect of the rate of heating on the alpha to gamma transformation temperature is also measured. It is shown that if the rate of heating above about 14,000°C per sec, it is possible to heat iron from the alpha to the delta phase, without any intermediate transformation into gamma.

Martensitic Phase in Eutectoid Cu-25 a/o Al Alloy: *Gareth Thomas* and *M. C. Huffstutler, Jr.*, University of California.

Transmission electron microscopy techniques have been used to study the structure of a highly martensitic Cu-25 at pct Al alloy. Comparison of direct micrographs and diffraction patterns indicated the presence of transverse (10$\bar{1}$2) twins in the lenticular β_1' plates. There is evidence that the structure is ordered, as expected for a transition from an ordered parent phase. Tempering experiments have been carried out in the microscope using a hot stage; unfortunately the cooling rate of the foils was not rapid enough to follow the $\beta_1 \rightarrow \beta'$ transition directly in the microscope.

Effect of Applied Tensile Stress on the Formation of Martensite in a Low Alloy Steel: *M. G. H. Wells*, Jones & Laughlin Steel Corp., and *D. R. F. West*, Imperial College of Science and Technology.

The course of the martensite transformation has been investigated by using an apparatus designed to measure simultaneously the temperature and electrical resistance of a thin wire specimen during continuous cooling. The apparatus had provision for the application of dead weight tensile loads at various temperatures.

Stresses causing both elastic and plastic strains have been found to affect the austenite to martensite transformation. A stress producing only elastic strain applied below the M_s temperature induces a burst of transformation as the load is applied. An indication of a small elevation of M_s was observed as a result of elastic strain of the austenite. Plastic deformation of the austenite in a limited temperature range above M_s induced a small amount of transformation to martensite while the specimen was deforming. The amount of induced transformation increased as the magnitude of the load increased, and the closer the temperature of application of a given load to M_s, the greater was the amount of martensite induced. Plastic deformation of the metastable austenite also mechanically stabilized the parent phase with respect to subsequent transformation on cooling, which is manifested by a depression of M_s. Appreciable plastic deformation of the specimen was observed in a small temperature range below M_s, when transforming under an applied stress, even for stresses well below the yield stress of the metastable austenite. It is suggested that this *transformation plasticity* is a result of preferred slip of the austenite adjacent to a growing martensite plate.

Surfaces

9:00 am to 12:00 m — **Shelby Room**

(Based on Submission of Research Abstracts)

Chairmen:

J. P. Hirth, Ohio State University, and *W. D. Robertson*, Yale University.

Effect of an Adsorbed Gas on the Anisotropy Surface Energy in Metals: *N. A. Gjostein*, Ford Motor Co.

To determine the effect that an adsorbed gas may have in altering the energy of surfaces near a low index orientation, it is assumed that gas atoms adsorb either at surface or ledge sites. The concentration of gas atoms in each kind of site is determined as a function of pressure, temperature, and the adsorption energies of the two types of sites. The Gibbs adsorption isotherm is used to relate the surface energy changes to these variables. The final expression quantitatively predicts the alteration in shape of an inward cusp in the surface energy polar plot. The implications that this effect may have on anomalous thermal grooving phenomena, and on faceting is discussed.

Effect of Orientation on Surface Diffusion in Copper: *J. Y. Choi* and *P. G. Shewmon*, Carnegie Institute of Technology.

The effect of crystallographic orientation on surface diffusion in pure copper was studied by means of thermal grooving of grain boundaries in bicrystals. Experiments were run with both 99.999 and 99.99 pct pure copper, but there was no effect of this variation of impurities on the surface diffusion coefficient.

The effect of orientation was studied on bicrystals containing symmetric tilt grain boundary form by rotation about [111], [110], or [100] directions. The surface diffusion coefficient, D_s, was determined on each of these three low index surfaces as well as on three other high index surfaces 90° away from these. Anneals were made in the range of 850° to 1050°C. For the low index planes the values of D_s were in the ratio D (111) : D (110) : D (100) : : 1.7 : 1.4 : 1. On the high index planes the most disordered surface (highest index) had the largest value of D_s but even here D_s was only twice the lowest value at that temperature. These ratios were essentially temperature independent, indicating that the activation energies for diffusion on all surfaces studied were about equal.

This work was supported in part by the US Atomic Energy Commission.

Measurements of Surface Diffusion Coefficients on Silver Single Crystals: *J. B. Drew* and *J. J. Pye*, Franklin Institute Laboratories.

Measurements of the surface diffusion coefficients of metals have been made. Diffusion profiles for the nickel-silver and silver-silver systems were obtained by means of a radioactive point source and a precision autoradiographic technique. The activation energy for silver self diffusion (approximately 6 kcal per mole) was lower than that previously reported (approximately 10 kcal per mole) on polycrystalline wire by Nickerson and Parker. The present data indicate an effect due to parasitic volume diffusion at temperatures above 500°C. The dependence of the diffusivities on surface macrostructure and surrounding atmospheres were investigated.

This work was supported by the United States Atomic Energy Commission.

Effect of Oxygen on the Evaporation Rate of Copper: *Robert Haverstraw*, Westinghouse Electric Corp., and *P. G. Shewmon*, Carnegie Institute of Technology.

It has been suggested by several investigators that the vapor pressure of Cu_2O is greater than the vapor pressure of copper. This leads to the expectation that pressures of oxygen, below that necessary to form bulk oxide on the surface of a sample, would nevertheless cause some copper to evaporate as Cu_2O thus increasing the apparent vapor pressure of copper.

Evidence has been obtained to indicate that the measured vapor pressure of copper remains substantially unchanged with increases in the oxygen pressure up to the amount necessary to form Cu_2O.

However, if the copper is deoxidized with hydrogen gas before evaporation is begun, the evaporation rate decreases by over an order of magnitude. This phenomenon can be reversed by subjecting the slowly evaporating sample to an atmosphere of one micron of oxygen pressure.

From this evidence it is proposed that in the usual experimental measurement of the evaporation rate of copper, dissolved oxygen in the sample diffuses to the surface and causes the sample to evaporate as Cu_2O.

Substantiating evidence for this hypothesis was found by measuring the evaporation rate of Cu_2O and noting that this value is identical to the value published for the evaporation rate of copper.

This work supported in part by the US Atomic Energy Commission.

THURSDAY, OCTOBER 26

Direct Observation of the Growth of Aluminum Oxide on Aluminum Single Crystals: *P. E. Doherty* and *R. S. Davis*, Arthur D. Little, Inc.

Aluminum oxide is produced on aluminum single crystals by heating to elevated temperatures in air. Small surface pits form under the aluminum oxide layer on cooling. These pits are attributed to the condensation of vacancies from a supersaturated solution. The oxide over the pits may be stripped off with a cellulose nitrate replica. The oxide particles adhere to the replica and may be examined directly in an electron microscope. It is shown that the oxide growth occurs at the oxide: metal interface; i.e., that the diffusion of oxygen through the oxide is the mechanism responsible for growth. Observations of the nucleation and growth of crystalline oxide at elevated temperatures shows that the orientation of the oxide is related to the orientation of the crystal surface on which it grows. In several cases, single crystal oxides were produced.

Silicon Ribbons and Filamentary Crystals: *E. S. Greiner, J. A. Gutowski*, and *W. C Ellis*, Bell Telephone Laboratories, Inc.

Small silicon ribbons have been prepared in a closed quartz tube in a temperature gradient in which iodine and hydrogen are initially present and in which a reaction occurs at 1100°C with silicon heavily doped with arsenic. Ribbons and needle-like crystals of silicon forms as a loose mass in the tube at about 950°C. The ribbons are as long as 2 cm and have widths as great as 0.15 mm. A range of thicknesses has been obtained; the thinner ones are semitransparent. The needle-like crystals are sometimes a centimeter long and have hexagonal cross-sections. R. S. Wagner and R. G. Treuting found the ribbons to be twinned crystals and the needle-like crystals to be single, as reported in detail in an accompanying abstract.

The transfer process may be more complex than simple disproportionation. The occurrence of ribbons depends upon the presence of arsenic and also upon small amounts of other added elements. Basic facts about the nucleation and growth of the deposits are discussed but the mechanisms are at present only partially understood.

Morphology and Growth Mechanisms of Silicon Ribbons and Whiskers: *R. S. Wagner* and *R. G. Treuting*, Bell Telephone Laboratories, Inc.

In an accompanying abstract, E. S. Grenier, J. A. Gutowski and W. C. Ellis describe the vapor growth of silicon crystals of two distinct forms: acicular and ribbon-like.

The acicular form is that of a whisker of hexagonal cross-section. The direction of growth is [111] and the whisker side faces are formed by the six {211} planes which belong to the [111] zone. It was found that every well-developed whisker contains a single dislocation parallel to the direction of growth, which appears to be essential for crystal growth.

The silicon ribbons are fairly uniform in width and very uniform in thickness. The widths range from 30 to 150 microns; the thicknesses range from 0.1 to 15 microns; the lengths range from 1 to 20 mm. The main faces of the ribbons are {111} planes, and the direction of growth is <211>. The ribbons are bounded on on their sides by {311} planes forming a ridge configuration. It was found that each ribbon contained one coherent twin plane parallel to the {111} main face. A mechanism of growth, based on the presence of this coherent twin plane, is suggested.

Transition From Internal to External Oxidation in Silver-Indium Alloys: *R. A. Rapp*, Wright-Patterson Air Force Base.

An alloy of silver which undergoes internal oxidation with low percentages of a less noble alloying element will form a compact, protective layer of oxide, exclusive of an internal oxide, when the mole fraction of the less noble component exceeds certain critical amounts. Wagner has proposed that this transition from internal to external oxidation with increasing alloying addition is due to a mechanical blocking of the diffusion processes caused by the precipitated oxide particles in the matrix. When the volume fraction of the oxide phase exceeds a critical mole fraction, further precipitation should occur in the same zone as the prior precipitation, and a compact oxide layer should ultimately result. To test Wagner's theory, 14 Ag-In alloys of N_{In} between 0.0093 and 0.163 were oxidized at 550°C and P_{O_2} between 10^{-4} mm Hg and 1 atm.

The transition from internal to external oxidation for Ag-In alloys oxidized at 550°C and P_{O_2} = 1 atm occurred for $N_{In} > 0.15$, i.e., if the In_2O_3 particles in the oxidized alloy exceeded 30 volume pct. For internal oxidation experiments at reduced oxygen pressures the supply of oxygen is less, and significant diffusion of indium outward, as well as oxygen inward, brings about an enrichment of indium in the internally oxidized zone over that in the bulk alloy. Solving the diffusion equations, the degree of indium enrichment for each reduced oxygen pressure was calculated. The N^*_{In} in the bulk alloy necessary to form a volume fraction $In_2O_3 = 0.30$ was then calculated for each reduced oxygen pressure. Metallographic investigation of the alloys oxidized at reduced oxygen pressures indicated that the transition occurred in agreement with the calculated values of N^*_{In}, and leads to the conclusion that the formation of 30 volume pct In_2O_3 in the Ag matrix is a valid criterion for the transition from internal to external oxidation in Ag-In alloys.

Recrystallization, Grain Growth, and Textures

9:00 am to 12:00 m — Sky Room

(Based on Submission of Research Abstracts)

Chairmen:

J. W. Rutter, General Electric Co., and *R. H. Richman*, Ford Motor Co.

Origin of Lineage Substructure in Aluminum: *Bruce Chalmers*, Harvard University, and *P. E. Doherty*, Arthur D. Little, Inc.

Using the Schuls X-ray and vacancy pit techniques for observing low angle boundaries, a study was made of the transition from the nearly perfect seed to the striated structure characteristic of large aluminum crystals grown from the melt. It was found that the individual striation boundaries develop by the coalescence of very small angle boundaries, as well as by the addition of individual dislocations. Evidence was found that a supersaturation of vacancies exists near a growing interface, and it is proposed that the resulting climb of existing dislocations produces half loops at the interface, which combine to form the low angle striation boundaries.

Grain Encounter Interactions in Polycrystalline States: *J. P. Nielsen*, New York University, and *L. P. Stone*, American Brass Co.

In two-dimensional polycrystalline states, grain encounters should occur when a four-ray junction occurs. Because grain boundary energies are not all equal a considerable variety of possibilities for the subsequent movements can be derived. These depend upon the distribution and degree of nonequilibrium that exists at the junction point for all the three-ray junctions that can be resolved at this point. In the two-dimensional case the analysis is academic because of the relative nonexistence of grain growth. In the three-dimensional case the encounters occur at five-grain junctions with a greater variety of possibilities, likewise depending upon the distribution and degree of nonequilibrium for the various four-ray junctions that can be resolved at a five-grain junction. In this case the analysis is significant in depicting grain boundary movements.

Grain Growth in High Purity Beta Brass: *L. P. Stone*, American Brass Co., and *J. P. Nielsen*, New York University.

Two beta brass (47.1 at pct Zn) ingots, 704 and 996 gm respectively, were cast from high purity copper and zinc (>99.999 pct) and processed into bars by repeated deformation and recrystallization. The large maximum grain size produced in these bars by normal grain growth at high temperatures for long times was considered as the qualitative indicator that the inclusion content of the bars was exceedingly low. The grain size attained after a vacuum anneal of 88 hr at 760°C (mp ~ 875°C) was 1.7 mm, 3.6 mm, 5.2 mm, and 14.1 mm in 28 mm diam disks which were 1.2 mm, 2.6 mm, 5.2 mm, and 9.5 mm thick, respectively. A similar disk, 9.3 mm thick and vacuum annealed for 284 hr at 490°C, attained a grain size of 5.7 mm and was still undergoing normal growth. Grain growth studies were made in the temperature range from 400° to 700°C with cylindrical specimens (12.3 mm diam x 12 mm long) having an initial average grain diam of 0.25 mm.

THURSDAY, OCTOBER 26

Employing the equation $D = kt^n_e$ (where t_e is the actual time-at-temperature), the values of n obtained were 0.29, 0.29, 0.29, 0.34, 0.31, 0.38, and 0.30 (averaging 0.314) at 395°, 447°, 497°, 552°, 600°, 682°, and 700°C, respectively. It is believed that these n values were obtained on grain growth that was 1) independent of free surface retardation effects, 2) relatively free, compared with previously reported data, of inclusion-controlled effects, and 3) free from discontinuous grain growth. The growth kinetics are shown to obey very closely the equation $D^2 - D_o^2 = K(t - t_o)$, where D_o is the average grain diameter (at time t_o) when the specimen reached the annealing temperature, over the entire range of temperature and grain size studied. In an Arrhenius plot the data yields a curved line. By approximating the curvature with a series of straight line segments, the values obtained (for the temperature ranges 400° to 500°, 500° to 600° and 600° to 700°C, respectively) in $K = K_o e^{-Q/RT}$ are: $K_o = 10^6 \frac{cm^2}{sec}$ and $Q = 44 \frac{Kcal}{g\text{-}atom}$, $K_o = 9 \times 10^{12} \frac{cm^2}{sec}$ and $Q = 33 \frac{Kcal}{g\text{-}atom}$, and $K_o = 4 \frac{cm^2}{sec}$ and $Q = 24 \frac{Kcal}{g\text{-}atom}$. It would appear that at the melting point the activation energy approaches a value close to zero.

Theory of the Formation of Rolling Textures in F.C.C. Metals: A Classical Approach and a Dislocation Interpretation: *Y. C. Liu*, Ford Motor Co.

In an attempt to provide a fundamental theory for the formation of rolling textures, a detailed analysis has been made of the flow mechanisms in fcc metals during rolling. The principal assumptions of this analysis are the following: 1) the stress field on an interior grain in the polycrystalline matrix is the same as the externally applied stress, i.e., a bi-axial stress with a tensile component parallel to the rolling direction and a compression component perpendicular to the rolling plane; 2) deformation occurs by slip on a {111} plane in a <110> direction; 3) both stress axes cause plastic flow in the same active slip directions; and 4) the equilibrium positions of the stress axes are the ideal orientations derived from a given flow mechanism.

By varying the number of active slip directions, and therefore, the number of active slip systems, the major ideal orientations proposed by various investigators can be explained and predicted. If eight active slip systems are considered, the ideal orientations derived from this mechanism are {135} <211>, and {8 13 21} <21 8 13>, and {4 5 7} <6 9 16>. The former is the orientation observed in AgCl and the latter are similar to the {358} <835> and {234} <323> orientations observed by Barrett and Steadman in copper. If only four active slip systems are considered, the ideal orientations derived are the {110} <112>, as in 70:30 brass, the {3 6 11} <13 8 8> and/or {8 13 21} <23 12 16>, as in aluminum, and the {110} <8 8 13> type, as in FeNi and Mo-permalloys.

Employing the analysis of Hirth for the interactions of dislocations in a fcc lattice, it is concluded that in the formation of preferred orientations all moving dislocations must interact to lower the total energy. In order to fulfill this condition, the flow mechanism for Cu-type texture can only be valid in metals in which the separation of two partial dislocations is less than five times the lattice parameter. This will explain why Pb exhibits a Cu-type texture and Ag does not, although the stacking fault energy in both metals is nearly the same (24 vs 21 ergs per sq cm, respectively).

Rolling Texture Formation in Aluminum-Iron Single Crystals: *Hsun Hu* and *R. S. Cline*, United States Steel Corp.

The development of rolling textures in two single crystals of a vacuum-melted 2 pct aluminum-iron alloy with initial orientations of (111) [$\bar{1}$12] and (112) [11$\bar{1}$] was studied in detail. These crystals were unidirectionally cold-rolled to 30, 50, and 70 pct reductions in thickness and their textures in the surface layer as well as in various sections through the specimen thickness were determined. In the (111) [$\bar{1}$12] crystal, no basic change in orientation was observed at the three reductions studied, which is consistent with the results for silicon-iron crystals reported in the literature. The texture becomes sharper on going from the surface layer down to the central section of the specimen. The orientation spread of the surface texture is practically the same at all three reductions.

In the cold rolled (112) [1$\bar{1}$1] crystal the surface texture consists mainly of two components, the (111) [$\bar{1}$12] and (001) [110], at all three reductions. The orientation spread of the surface textures here appears also to be independent of the amount of reduction. However, the relative intensity of the two components varies with the amount of reduction. At 30 pct reduction, the two components are of approximately equal intensity. With increasing reduction the (111) [$\bar{1}$12] component becomes stronger whereas the (001) [110] component becomes weaker.

The relative amount of the two components varies also with the depth below the specimen surface. The (001 [110] component diminishes towards the central section of the specimen. At all three reductions the texture close to the central section of the specimen is a single (111) [$\bar{1}$12]. With a 3.25 pct silicon-iron alloy, Koh and Dunn obtained approximately equal amounts of (111) [$\bar{1}$12] and (001) [110] components from the central section of a (112) [1$\bar{1}$1] crystal cold-rolled 70 pct. The difference between Koh and Dunn's results and the results of the present investigation may be due to the manner of rolling.

Surface Energies and Other Surface Effects Relating to Secondary Recrystallization Textures in High-Purity Iron, Zone-Refined Iron, and 0.6 pct. Silicon Iron: *C. G. Dunn* and *J. L. Walter*, General Electric Co.

A previous report described the occurrence of secondary recrystallization to the (110) [001] texture in high-purity iron annealed in purified hydrogen. Further studies that included zone-refined iron and a high-purity silicon iron containing 0.6 pct Si have been made. These studies consider the effects of annealing atmospheres and other variables on the driving forces that control the development of different secondary recrystallization textures through control of (hkl), surface energies.

The high-purity iron and 0.6 pct silicon iron were melted and cast in vacuum. The zone-refined iron was prepared from spectroscopically pure iron. Samples were rolled to 0.003 to 0.012 in.

In vacuum anneals, secondary recrystallization, to the (100) [001] texture, was obtained in zone-refined iron (890°C) and in 0.6 pct silicon iron (925°C). In dry hydrogen, growth of either (100) or (110) secondaries (and under certain conditions, of both (100) and (110) secondaries) was obtained. Thus, in slightly oxidizing atmosphere (vacuum), γ_{100}, the energy of the (100) surface, was lowest. In dry hydrogen γ_{110} was the lowest or became lowest by removal of oxygen during the anneal.

The primary recrystallization texture of the 0.6 pct silicon iron consisted of two (111) [110] components and a (111) fiber component, whereas the texture of similarly prepared high-purity 3 pct silicon iron consisted largely of a moderately strong (110) [001] component.

An effect of surface oxygen on grain growth inhibition during vacuum anneals of high-purity iron was observed. Inhibition, beyond that caused by the thickness effect, did not occur in anneals in dry hydrogen. The presence of silicon reduced the inhibiting effect of oxygen without altering the (hkl), relationships.

Some information on annealing twins and on oxidation is presented.

X-Ray Methods for Pole Figure Determination and Quantitative Phase Analysis in Textured Materials: *S. L. Lopata* and *E. B. Kula*, Watertown Arsenal.

A modification is shown of the Schulz reflection method for determining pole figures which avoids the use of a separate transmission sample. A surface whose normal is equidistant (54° 44') from the rolling plane normal, rolling direction, and transverse direction is cut on a composite sheet or solid sample. Complete information for one quadrant of the pole figure can be obtained by diffracting from this surface, with the geometry of the Schulz reflection method being maintained. A polar stereographic net has been constructed for use in plotting data from a sample surface cut at this angle. In addition, a randomizing net is shown which allows determination of the random intensity level of a pole figure, thereby obviating the necessity of experimentally determining the random intensity on a separate random sample.

Also shown is a method for quantitative phase determination in textured materials. This is done by moving the specimen in the X-ray beam both physically and mathematically so that the area under a diffraction peak is integrated over the entire pole figure. Two methods of doing this are discussed. In materials with a fiber axis, a surface is cut 45° to the fiber axis and the intensity is measured as the angle α between the fiber axis and the diffracting plane normal is varied from 0° to 90°. In rolled material the specimen is rapidly rotated around the sheet normal and diffracted intensity is recorded as the angle α between the sheet normal and diffracting plane normal is varied. Separate reflection and transmission samples are required in this case, however. For both cases graphic integration of a plot of I sin α versus α will yield the total diffracted intensity of each phase. This information can then be used to find the volume fraction of each phase.

THURSDAY, OCTOBER 26

Subgrain Coalescence and Recrystallization of a Silicon-Iron Crystal as Observed by Transmission Electron Microscopy: *Hsun Hu*, United States Steel Corp.

The early stages of recrystallization in a 3 pct Si-Fe crystal with an initial orientation of (110) [001] after 70 pct reduction by cold rolling were studied by electron transmission microscopy. Observations were made with thin foils prepared from the variously annealed bulk specimens, and also with thin foils from the as-rolled crystal which were then annealed by means of a hot stage inside the microscope during examination. It was found that when annealed as thin foils, polygonization takes place rapidly at 600°C (although a much lower temperature is required in bulk specimens), followed by a slow increase in the average subgrain size on heating to higher temperatures, before recrystallization can be detected. The growth of subgrains appears to proceed largely by gradual disappearance of some of the subgrain boundaries, i.e. by the *coalescence* of subgrains. Such coalescence can be explained by the moving-out of dislocations from the disappearing subgrain boundaries into the connecting boundaries around the subgrains. The angular misfit of the connecting boundaries may thus become higher. This process probably involves dislocation climb along the disappearing boundary, together with rotation of the subgrains which requires movement of some of the atoms around them. The formation of recrystallization nuclei by migration of high-angle boundaries attached to the initial subgrains was not observed. A recrystallized grain at the earliest stage of formation that could be detected by electron diffraction was found to consist of a group of coalesced subgrains, and only segments of its boundary appeared to be of the high-angle type.

This evidence suggests that recrystallization nuclei are probably formed by *coalescence* of subgrains. Based on this model many features of the recrystallization process, such as the incubation period, and the dependence of the recrystallization tendency of a deformed crystal upon sharpness of its deformation texture, upon size of its polygonized subgrains, and upon the temperature of deformation, etc., can be reasonably explained.

Subgrain Growth by Subgrain Rotation: *J. C. M. Li*, United States Steel Corp.

The process of subgrain growth by rotation of a subgrain so it can coalesce with one of its neighbors, as suggested by Hu in the previous abstract, is analyzed thermodynamically and kinetically. It is found that the process is thermodynamically possible if all the neighboring boundaries decrease their angles of misfit, or if the low-angle boundaries decrease their angles of misfit while the high-angle boundaries *increase* their angles of misfit. The second possibility helps subgrain growth by creating more high-mobility boundaries. The rate of rotation of a subgrain with respect to its neighbors is estimated using a dislocation model in which the dislocations climb cooperatively and using an atomistic model in which the atoms diffuse all around the rotating subgrain, also cooperatively. It is found that both models indicate that the rate of rotation varies inversely with the cross-sectional area of the rotating subgrain perpendicular to the rotating axis, and proportionally with the driving force of rotation and self diffusivity. The estimated rate of rotation is consistent with some indirect experimental evidence.

Effect of Temperature on the Rolling Textures of Molybdenum: *J. R. Holland*, United States Air Force.

Commercial purity arc-cast molybdenum, extruded at 1093°C., was reduced from 90 to 94 pct by rolling at temperatures in the warm work and hot work range (from 760° to 1550°C). The textures of the rolled strip were determined by quantitative X-ray techniques, and as rolled hardnesses and microstructures were determined. The effects of rolling temperature on textures has been established. The effect of strain rate, total reduction, and intermediate annealing times (between roll passes) have been evaluated. The (100) 011 and (211) 011 orientations are stable components of the rolling textures over a wide range of rolling conditions. The texture results on molybdenum are compared with other bcc metals. Possible explanations of the observed hot rolling textures are offered. The warm rolling textures of molybdenum are compared to the predictions of Calnan and Clews.

The effects on the chemistry of warm rolling and hot rolling molybdenum in air have been investigated. Decarburization was noted, but there was no apparent increase in the content of other interstitials. The chemical analyses were determined spectrographically.

Solidification

2:00 to 5:00 pm — **Terrace Room**

(Based on Submission of Research Abstracts)

Chairmen:

R. S. Wagner, Bell Telephone Laboratories, Inc., and *H. W. Weart*, Cornell University.

Determination of the Atomic Kinetics of the Freezing Process: Part I: *W. A. Tiller* and *J. J. Kramer*, Westinghouse Electric Corp

Theoretical treatments of the atomic kinetics of the freezing process have related the interface velocity to the departure from equilibrium at the interface for several different atomic mechanisms. At the present time no general experimental method for assessing these relationships exists in the literature for the case of crystals grown from the melt. Such information is invaluable to a proper assessment of the various mechanisms that apply to a particular class of materials (i.e., metals, semiconductors, oxides, etc.).

The authors have invented a new technique which appears to have general applicability and which is capable of testing the various theories. It consists of subjecting the solid-liquid interface to a planar harmonic thermal wave and measuring the attenuation of the wave across the solid-liquid interface.

This paper is a theoretical assessment of the method. It is found that, from the response of the interface to the thermal wave, the atomic kinetics of the freezing, and melting processes can be explicitly evaluated.

Solute Concentration at a Cellular Interface During Solidification: *J. J. Kramer, G. F. Bolling*, and *W. A. Tiller*, Westinghouse Electric Corp.

The steady-state solute concentration in the liquid at a smooth planar interface has been shown to be given by C_∞/k, where C_∞ is the concentration of the bulk liquid and k is the solute partition coefficient. For a cellular morphology this interface concentration can be defined as $\alpha\ C_\infty/k$, where α depends upon the cell size and the growth variables. This parameter α is very important to our theoretical understanding of the cellular interface morphology and determines the degree of chemical segregation occurring at the cell boundaries.

A theoretical assessment of the value of α has been made and has been found to coincide very closely with experimentally determined values for the Sn-Pb system.

The experimental technique involves the measurement of the interface temperature at the cell caps by mans of a thermocouple probe. The liquid concentration at the cell caps is simply related to the cap temperature.

The application of the thermocouple probe technique as a tool in studying solidification phenomena will also be discussed.

Fluid Flow Control During Solidification–Part II: Programmed Solute Distribution: *W. C. Johnston* and *W. A Tiller*, Westinghouse Electric Corp.

One of the objects of controlled solidification is to utilize the freezing process for the preparation of crystals of specific materials containing a predetermined distribution of solutes.

A theoretical analysis of alloy solidification in which the effective partition coefficient, k, of the solute is allowed to vary as a function of interface position has been performed. A variety of solute distributions in the solid are predicted depending upon the functional dependence of k with interface position. Conversely, the variation of k with interface position required to produce a particular solute distribution in the solid can be calculated. These calculations apply both to a single-pass-zone melting process as well as a normal-freeze-process.

These predictions have been tested by controlling the momentum boundary layer as a function of interface position by programmed magnetic stirring. Data obtained by programmed magnetic stirring of lead-tin alloys during normal freezing are presented. Both a linear and a cosine solute distribution in the ingot have been produced for high and low solute concentrations in agreement with the theoretical predictions.

THURSDAY, OCTOBER 26

Structure of Alloys Rapidly Cooled From the Melt: *Pol Duwez, R. H. Willens,* and *W. Klement, Jr.,* California Institute of Technology.

By ejecting small molten droplets onto a good thermal conductor, it is possible to obtain a thin liquid layer which is then cooled extremely rapidly. For many alloys, the rate of cooling is sufficiently great so as to forestall the normal nucleation and growth of the equilibrium phases. Complete solid solubility, as suggested by the Hume-Rothery criteria, has been obtained in Ag-Au, Ag-Pt alloys as well as for the pseudo-binary system GaSb-Ge. Investigation of the homologous systems Ag, Au-Ge, Si has yielded strikingly dissimilar results. In Ag-Ge, the primary solid solubility has been increased to approximately 13 at pct Ge; from approximately 15 to 20 pct Ge, an hcp phase of nearly ideal axial ratio predominates. The appearance of hcp lines in the fcc region, and vice versa, suggests that stacking faults are present but verification has not been possible because of the sub-micron grain size. In Ag-Si, the primary solid solubility is probably extended beyond the reported value but there is no detectable change in the lattice parameter. Hcp lines are found in the predominantly fcc alloys but no predominantly hcp alloy has been discovered—again suggesting stacking faults. In Au-Ge alloys, an hcp structure with Au or Ge is observed in the regions 0 to 25 at pct, 65 to 100 at pct Ge but, at the intermediate compositions, very complex structures are found. The grain size in the 42 at pct Ge alloy is much larger than has been observed in other alloys quenched under similar conditions. In Au-Si, an amorphous, liquid-like structure has been retained in the 25 at pct Si alloy. Unstable at room temperature, this amorphous structure decomposed within hours into a complex crystalline structure involving d-spacings approximately 11 to 12 Å.

Growth From the Melt III: Dendritic Growth: *G. F. Bolling* and *W. A. Tiller,* Westinghouse Electric Corp.

A mathematical treatment of steady-state dendritic growth from pure and alloy liquids has been developed for isothermal and non-isothermal dendrites. The velocity of dendritic growth, dendrite size, dendrite direction and the alloy content of the dendrite have been calculated as a function of the bath supercooling, the alloy content of the bulk liquid and the atomic kinetics of the material. To completely specify the problem it was necessary to choose some *optimization* condition. We have chosen the condition of *maximum velocity*. Comparison of the theoretical predictions with experimental data for pure nickel, tin, and ice and for KCl and acetic acid solutions have been made. Satisfactory agreement with experiment was found using the atomic mechanism of *uniform interface advance*.

Preferred Orientations Developed During the Solidification of High Purity Lead: *G. F. Bolling, J. J. Kramer,* and *W. A. Tiller,* Westinghouse Electric Corp.

The orientations developed during the solidification of zone-refined lead have been examined. This was done by using a novel casting technique having several advantages over those previously used; unidirectional heat flow and good atmosphere control were possible and an excessive use of high-purity material was avoided. It was also possible to decant the liquid at any stage of growth in order to observe the solid-liquid interface.

The primary observations were the existence of both a <111> growth texture in the columnar zone of the ingots and a (111) texture at the chill surface at the mold wall. Several variations of the basic casting experiment such as changing the oxygen content of the lead and changing the shape of the mold wall were carried out. The results of all the experiments support the separate existence of both a preferred (111) nucleation and a preferred <111> growth direction in zone-refined lead. The conditions of casting were not conducive to dendritic growth and no evidence for this type of growth was found at the solid-liquid interface or in the structure of the columnar grains. Several observations of the interface were made, all revealing a platelet structure related to the (111) planes.

Several theories which might predict preferred orientations are discussed and observations in pure tin and pure zinc are briefly mentioned.

Solidification of Dilute Binary Alloys: *F. Weinberg,* Department of Mines and Technical Surveys, and *E. H. McLaren,* National Research Council (Canada).

Dilute binary alloy ingots have been solidified under controlled thermal conditions and the solute distribution, temperature during freezing, and the position and morphology of the solid-liquid interface have been examined. The binary alloys investigated were Pb, Sn and Zn based alloys with solute additions of Ag^{110}, Au^{198}, Sb^{124}, Tl^{204}, Sn^{113} and Zn^{65}. Approximately 150 cu cm of metal was used in each ingot.

It was found that appreciable solute segregation occurred in most of the ingots, depending on the solute concentration, distribution coefficient, freezing rate, and the purity of the alloy elements. Measurements were made of the general solute distribution by sectioning layers of material from the ingot circumference and counting the activity of each layer. Local segregation was detected by autoradiographic techniques.

The temperature of the alloys during freezing was determined by precision resistance thermometry. Since both freezing curves and solute distribution curves were obtained for each ingot, the effect of solute segregation on the shape of the freezing curve could be determined. It was found that the freezing curve was significantly affected both by solute segregation and the configuration of the solid-liquid interface during freezing.

The position and morphology of the solid-liquid interface was determined in the Zn based alloy for slow growth rates. This was done in two ways: by decanting the liquid during solidification and examining the position and shape of the exposed interface, and by rapidly quenching the alloy during solidification. In the latter case the position of the interface at the time of quenching could be determined from autoradiographs of the sectioned surfaces of the ingot, and from etch patterns on these surfaces.

The results are considered in relation to current theories of the solidification process in dilute binary alloys.

Solidification of $CuAl_2$-Al Eutectic Alloys: *G. A. Chadwick,* Harvard University.

The variation of micromorphology with growth conditions has been investigated over a range of growth rates from 100 to 0.2 cm per hr using eutectic alloys made from slightly impure and zone-refined metals. The relationship between the interlamellar spacing and the rate of solidification has been established quantitatively. Several approaches have been used to determine the slope of the solid-liquid interface under equilibrium conditions and during steady state growth.

Crystallography of Equilibrium Phase Interfaces in Al-$CuAl_2$ Eutectic Alloys: *R. W. Kraft,* United Aircraft Corp.

Using a new X-ray diffraction camera, orientation data were obtained on the crystallites of both phases in several slowly grown eutectic grains, each of which consisted of a nearly perfect parallel arrangement of alternating platelets of Al and $CuAl_2$. Angular measurements of the intersections of the lamellae with two surfaces of the specimens provided a means for measuring and describing the interfaces between the phases since the bulk of the interphase boundaries were parallel to the lamellae. The growth direction was also know since the specimens were unidirectionally solidified. A consistent relationship was discovered among the orientation of the Al unit cells, the $CuAl_2$ unit cells, and the (lamellar) interfaces. Since knowledge of these three variables related to the same coordinate system is sufficient to completely specify all five degrees of freedom of an interface between two crystals, it was possible to analyze in detail the crystallography of what are presumed to be equilibrium Al-$CuAl_2$ interfaces.

The results of this work indicate that an equilibrum interface between Al and θ phase can be described in general as follows:

Interface || (111) Al || (211) θ
<101> Al || <120> θ

As far as the aluminum phase is concerned, the fact that the close-packed planes form the interfaces requires no further elaboration. A theory is advanced to explain why (211) planes of θ (which has a rather complex body centered tetragonal structure) are important low energy planes in these alloys. The essence of the argument is that a crystal of θ can be considered to be composed of planes with a stacking sequence, parallel to (211), described as ABCDABCDA . . . where each letter refers to a group of closely-spaced planes of atoms. It is thought that a group of planes can be considered as a *puckered* plane, and it is shown that the *puckered* plane can have precisely the same atomic density as the adjoining octahedral planes of Al simply by the mechanism or omission of a few atoms within the puckered plane. This model of the phase interface satisfactorily explains the rotational relationship of the two crystals about an axis perpendicular to the interface (second statement above) by a consideration of *pseudo* close-packed directions within the puckered plane. In addition, all features of the experimentally observed growth directions were satisfactorily accounted for.

In the course of developing this model, it was necessary to study the structure of $CuAl_2$ in detail. $CuAl_2$ is the prototype

THURSDAY, OCTOBER 26

of the C16 type structure, space group I4/mcm, with x = 0.16. Based upon the observation that (211) are low energy planes, a new concept is proposed to help explain why x in the C16 compounds tends to a value of 1/6 as has been observed in most other compounds with the same structure.

Interface Morphology of Magnesium-Aluminum Eutectic: *A. S Yue,* The Dow Chemical Co.

The interface morphology of high-purity magnesium-aluminum eutectic has been studied in terms of the freezing rate and the temperature gradient. It has been found experimentally that for a given temperature gradient a fast freezing rate favors the formation of a cellular interface consisting of sub-grain structures. It can be changed to a planar interface of the same sub-structures either by decreasing the freezing rate or by increasing the temperature gradient. Furthermore, a layer of liquid adhering to the interface during decantation indicates that shapes and sizes of the phase particles observed on the interface were different from those in the main eutectic solid.

Quantitative Metallography of Anisotropic Structures. Substructure of Lamellar Eutectic Alloy: *R. W. Kraft, F. D. Lemkey,* and *F. D. George,* United Aircraft Corp.

By a consideration of the ways in which an array of lines (or linear features) in three-dimensional space can depart from a statistically random or isotropic arrangement, a system is evolved to describe different types of anisotropy. Formulae are then developed by which the average number of intersections of the array of lines with sectioning planes, $\overline{N}_A$, is related to the type of anisotropy and to two angles, θ and ω, which define the orientation of section planes in the sample. Given sufficient data of the type $\overline{N}_A$ (θ, ω) it is therefore possible, in principle, to deduce the type of anisotropy and to calculate parameters which express the severity of the anisotropy.

The technique is then applied to a unidirectionally solidified lamellar eutectic alloy which contains lamellar faults in an anisotropic array. The type of anisotropy is deduced by fitting the experimental data to various trial equations and determining which gives the best statistical fit; the parameters of the *best* equation then described the severity of the anisotropy. A quantitative picture of an *average subgrain* which is far from isotropic is thereby obtained.

Precipitation and Ageing

2:00 to 5:00 pm — **Ballroom**

(Based on Submission of Research Abstracts)

Chairman:

J. B. Clark, Ford Motor Co.

Transmission Electron Microscope Investigations of Age-Hardening Systems: *Gareth Thomas,* University of California.

Thin foils of Al-Mg-Si and Cu-Be have been prepared by electropolishing and examined in the electron microscope after various stages of aging. Needle-shaped zones are observed in Al-Mg-Si which change continuously into rods and then plates of Mg_2Si with prolonged aging. In Cu-Be alloys small precipitates are observed at aging temperatures approximately 130°C around which contrast effects may indicate the presence of coherency strain fields.

A movie has been made of precipitation in Al-4 pct Cu alloys using a high temperature stage in the electron microscope. From measurements of the rates of solution of $CuAl_2$ particles it is possible to estimate the diffusion coefficient of Cu in Al.

Kinetics of Formation and Reversion of GP I Zones of Al-1.7 at pct Cu: *Herbert Herman,* Atomics International, and *M. E. Fine* and *J. W. Kauffman,* Northwestern University.

A study was made of kinetics of reversion and subsequent reformation of the planar and non-ideal GP I zones in Al-1.7 at pct Cu. Electrical resistance was the major property measured.

GP I dissolves in less than 0.5 min during a 205°C reversion treatment. The time for complete reversion is not dependent on the extent of primary aging. This independence of the reversion time on zone size is in line with the idea that GP I grows with constant thickness and must revert sideways. The total resistance change and the sign of the change vary with aging time but the resistance after reversion is constant.

After reversion the zones reform on aging near room temperature at rates much slower than after the quench. When care is taken in handling well-aged and reverted specimens an incubation time is noted before zone formation. The rate of reaging after reversion depends on the primary treatment prior to reversion decreasing with the extent of primary aging. Cold working after reversion has the effect of removing the incubation time and accelerating the reaction.

Reverted specimens were irradiated with 1 Mev electrons at near 77°K with a total flux of 6×10^{17} per sq cm. There was a 0.5 pct resistance increase at 77°K which seems to indicate that motion of interstitials also accomplishes zone formation. The incubation time was removed and the rate of reaging after reversion and irradiation is roughly that predicted from the concentration of vacancies which were thought to be present.

It is suggested that zone nucleation in Al-Cu is probably heterogeneous, and that an imperfection structure is associated with the zone.

This research was supported by the Air Force Office of Scientific Research under Contract No. AF 49(638)-524, and also in part by the Atomic Energy Commission.

Kinetics of Formation and Reversion of GP Zones in Al-5.3 at pct Zn: *Herbert Herman,* Atomics International, and *J. B. Cohen* and *M. E. Fine,* Northwestern University.

The kinetics of reversion and reaging after reversion in Al-5.3 at pct Zn has been studied using electrical resistance. The sizes of the Guinier-Preston zones were determined by small angle scattering.

Kinetics of reversion at 150°C was studied as a function of primary treatment, and it was found that the greater the extent of aging, that is, the larger the zones, the longer is the time required for complete reversion.

For reaging after reversion there is an effect of temperature of reversion. A retardation of kinetics was noted as the reversion temperature is decreased and the reaging temperature is raised (taking normal thermal variation into consideration). It is suggested that the retardation effect is due to the existence of a spinoidal line within the two phase region of the phase diagram.

X-ray scattering at small angles was used to measure zone sizes for direct-aging after the quench and reaging after reversion. Of particular interest was finding sizes at the peak in resistance which occurs during zone formation. It has been suggested by others that the resistance maximum is associated with a definite zone size. This has been confirmed and the critical diameter has been found to be approximately 10Å.

This research was supported by the Air Force Office of Scientific Research under Contract No. AF 49(638)-524.

Short Range Ordering vs. Solute Clustering in Alpha Brass: *H. M. Otte,* RIAS.

On aging at 120°C two annealed alpha brasses containing 30 and 35 pct Zn respectively, a small (ca 0.0003 to 0.0006 A) reduction in the lattice parameter occurs, which is generally attributed to an increase in short range order. In the present work all diffraction lines in the spectrum were measured using monochromated Cu $K\alpha_1$ radiation, and all indicated the same change. Cold work induced by filing produced a measurable amount of stacking faults as detected by the shifts in the diffraction lines and in addition caused an increase in lattice parameter of about 0.0008 A. On aging these cold worked alloys for over 10000 hr at 120°C, the lattice parameter dropped. For the Cu-35 Zn the drop was 0.0037 A and was accompanied by the precipitation of a small but measurable amount of beta phase. Thereby the solubility limit of the alpha phase was placed between 33 and 34 pct Zn. The Cu-30 Zn alloy also showed a drop in the lattice parameter of 0.0029 A but, as expected, no beta phase. In neither case was there any observable change in the stacking fault density. These data are interpreted in terms of solute segregation, possibly to stacking faults, as suggested by Suzuki. Solute clustering may also be the probable cause of the drop in lattice parameter for the annealed and aged alloys, rather than short range ordering. Supporting evidence for this was obtained from a Ag-25 Zn alloy, which, after quenching and aging for over 1000 hr at 120°C, showed a small increase in lattice parameter. Unless the alloy disorders under these conditions, which is improbable, this can only be explained by a clustering of the Zn atoms.

THURSDAY, OCTOBER 26

Discontinuous Precipitation in Binary Alloys of Alpha-Iron: *E. Hornbogen*, United States Steel Corp.

Discontinuous precipitation can occur if nucleation of a new phase in the matrix has a high activation energy. This is usually so if phases are precipitated that differ greatly from the matrix in specific volume or crystal structure.

Discontinuous precipitation requires the production of a site of lower activation energy by an autocatalytic reaction front that moves through the crystal. Three typical examples are described; Fe-24 pct Zn, Fe-12 pct Au and Fe-26 pct Mo alloys.

1) Fe-Zn—The common type of discontinuous precipitation, which starts on high angle boundaries only. This results in a lamellar structure growing into the homogeneous solid solution, with recrystallization of the alpha Fe in the aggregate.

2) Fe-Au—The mechanism is the same as in (1) but the product is not strictly lamellar. The reaction front does not move into a homogeneous solution, but into a fine dispersion of a non-equilibrium phase which is dissolved.

3) Fe-Mo—The reaction starts on existing dislocations and generates new dislocations. There is no recrystallization of the matrix and no motion of a high angle boundary. The reaction leads to a fine dispersion of non-lamellar particles.

The mechanisms of the three reactions are analyzed and the conditions under which the different kinds of discontinuous precipitation can be expected in alpha Fe are discussed.

Electron Transmission Study of Nitride Precipitation in Alpha Iron: *A. S. Keh* and *H. A. Wriedt*, United States Steel Corp.

The precipitation of nitride particles in alpha iron was studied by transmission electron microscopy.

Vacuum-melted iron annealed in wet hydrogen was used as the starting material. Specimens were first treated to give different dislocation structures. Then nitrogen between 0.005 to 0.040 wt pct was introduced using ammonia-hydrogen mixtures. Specimens quenched with the nitrogen in solution were aged at 25°, 100° and 200°C.

After aging, the precipitated nitrides, tentatively identified as $Fe_{16}N_2$ have {100} habit planes. Nucleation takes place mostly at dislocations and at sub-boundaries; some occurs in the matrix. But nucleation on high angle boundaries was not observed.

With respect to nucleation at dislocations and at sub-boundaries, an effect of aging temperature was observed. At room temperature, there is no particular orientation relationship between the dislocations involved and the {100} plane on which the nitride formed. However, with increasing temperature, the nucleation sites tend to become restricted to those dislocation segments lying in {100} planes; and on these {100} planes the precipitates subsequently formed. Nucleation in the matrix was observed only at 100°C or below in specimens with 0.02 pct N having very few dislocations (approximately 1 x 10^8 per sq cm) and no substructure. Nucleation in the matrix is believed to take place on vacancy clusters, or possibly on dislocation loops formed by vacancy condensation.

The change of yield stress of the 0.02 and 0.04 pct N alloys during quench aging at 100°C is correlated with the change of nitride distribution.

No precipitates can be seen on dislocations after strain aging.

Precipitation of Hydrogen in Iron-Silicon Single Crystals and the Mechanism of Hydrogen Embrittlement: A. S. *Tetelman*, Physique de Solides, (France) and *W. D. Robertson*, Yale University.

The technique of decorating dislocations with carbon has been employed to study the formation and growth of cleavage cracks produced by the precipitation of hydrogen in iron 3 pct Si single crystals. Hydrogen was introduced either by quenching from a hydrogen atmosphere in the range of 700° to 1200°C, or by the electrolytic reduction of hydrogen ions from sulphuric acid. The resulting embrittlement was investigated at room temperature and at −180°C.

Cleavage cracks on {100} planes are produced when single crystals are quenched from a hydrogen atmosphere and the total length of cracks inside the crystals was shown to depend on the hydrogen concentration. Identical cracks are formed in annealed crystals by cathodic charging and their number depends on current density. The detailed characteristics of plastic deformation in the vicinity of cracks were observed in terms of decorated etch pits and the deformation was shown to be defined by crystallography and to be consistent with the calculated stress distribution about a crack growing under internal pressure.

Fracture and embrittlement phenomena in single crystals were studied under conditions of static and dynamic tensile loading and the results are interpreted in terms of the observed growth of preexisting cracks produced by the precipitation of hydrogen gas.

This investigation was sponsored by the Office of Naval Research.

The Effect of Prestraining and Aging on the Tensile Properties of a High Strength Steel: *S. C. Das Gupta*, *P. J. Fopiano*, and *S. A. Kulin*, Manufacturing Laboratories, Inc.

The effect of prestraining and aging treatments on the subsequent tensile properties of heat-treated vacuum melted H-11 been investigated. Electron microscopy, X-ray diffraction, and fracture toughness have also been employed in the evaluation of the observed tensile property changes.

The yield and ultimate tensile strengths are found to increase with increasing prestrain (between 0.5 and 4.0 pct) and to have maximum values for aging temperatures in the range of 600° to 800°F. The position of this maximum shifts to lower aging temperatures for higher values of prestrain. In addition, variation of aging time at constant values of prestrain indicate the possible presence of two maxima in the stress increment as a function of aging time.

An unexpected yield point effect is observed for prestrained and aged specimens of this high-strength steel. The magnitude of this effect increases with higher values of prestrain and higher aging temperatures and attains a maximum at a temperature of about 800°F.

Changes in microstructure, as observed by electron microscopy, suggest that redistribution of carbides occurs as a result of the prestraining and aging treatments. The magnitude of the observed tensile property changes is apparently associated with the amount of such carbide redistribution.

Effect of Deformation Temperature Variations on the Strain Aging of Alpha-Iron: *E. Lautenschlager*, Allis-Chalmers Manufacturing Co.

Yield point measurements were made on polycrystalline specimens of α-iron deformed various amounts at 77°, 195°, and 273°K; aged; and redeformed at 77°, 195°, and 273°K. When the deformation temperature before aging is lower than that after aging, there is a suppression of the returning yield point with respect to specimens which were deformed at the same temperature both before and after aging. The degree of suppression is a function of both the amount of strain at the lower temperature and the difference between the deformation temperatures before and after aging. It is proposed that the suppression is caused by the formation of barriers during the initial low temperature deformation.

The aging kinetics of polycrystalline α-iron wires deformed at 77° or 303°K were studied at 44° and 64°C using internal friction measurements. No change was noted as a result of the difference in initial deformation temperature.

This work formed part of a thesis submitted by E. Lautenschlager in partial fulfillment of the requirements for the degree of Master of Science. This research was partially supported by the U. S. Air Force through the Air Force Office of Scientific Research of the Air Research and Development Command.

Precipitation-Hardening in Columbium Alloys: *J. E. McNutt* and *G. D. Gemmell*, E. I. du Pont de Nemours and Co.

Susceptibility to age-hardening is imparted to a broad range of columbium-base alloys by additions of zirconium. This has given a new path for development of high-temperature alloys, in which heat treatment plays a part in controlling strength and ductility at various stages of processing, independent of working and recrystallization.

Evidence is presented on the part that interaction of zirconium with interstitials in solid solution plays in formation of second phases. Details are given on times and temperatures of solution treatment and aging to develop various combinations of mechanical properties, up to 2400°F. Metallographic studies are reviewed, including electron microscopic and diffraction results. The effects of other elements on the thermodynamics and kinetics of the aging reaction are described.

Strain Aging in Columbium and Its Alloys: *J. E. McNutt*, E. I. du Pont de Nemours and Co.

Strain aging effects have been observed in columbium-base materials over the temperature range 1000° to 2400°F and the range 0.005 to 0.2 per min in strain rate. It seems clear that the same physical effects cannot give rise to occurrence of an upper and lower yield point at 1000° and 2400°F, at a strain rate of 0.05 per

THURSDAY, OCTOBER 26

min, for example. The available data are presented and discussed. No satisfying rationalization of these effects is available.

Structure and Properties

2:00 to 5:00 pm — Shelby Room

(Based on Submission of Research Abstracts)

Chairmen:

J. C. Shyne, Stanford University, and *W. L. Grube*, General Motors Corp.

Observations of Martensite Formed From Cold Worked Austenite I: Metallurgical Variables: *W. M. Justusson, D. J. Schmatz*, and *V. F. Zackay*, Ford Motor Co.

The strength of martensitic steels in which the martensite is formed from cold-worked austenite is greater than the same steels conventionally heat treated. The strength is observed to increase with increasing amounts of deformation, at a given deformation temperature; and, for a given amount of deformation, to increase with decreasing temperature of deformation. The increase in strength, for a given temperature of deformation and for a given amount of deformation, appears to be constant over a wide range of carbon content. The mechanical properties of these steels are unusually insensitive to tempering temperature. The yield strength and elongation are virtually constant for low and medium tempering temperatures. Unlike conventionally tested martensitic steels prior austenitic grain size appears to exert a negligible influence on the mechanical properties.

Observations of Martensite Formed From Cold Worked Austenite II: Strengthening Mechanisms: *D. J. Schmatz, W. M. Justusson*, and *V. F. Zackay*, Ford Motor Co.

Several strengthening mechanisms are discussed which are considered to be operative in steels in which the martensite is formed from cold-worked austenite. These are: inheritance of defects by the martensite from the austenite; altered martensite morphology; enhanced precipitation hardening; and the development of a martensitic fine structure.

The yield strength of a very low carbon severely deformed austenitic Fe-Ni alloy was found to be indentical to that of a fully-martensitic structure whether formed from either a cold-worked or an annealed austenitic matrix. This is in accordance with a suggestion of Kurdjumov that the strengthening observed in the martensitic transformation of a low carbon iron is equivalent to that obtained by severe deformation. The morphology of martensite formed from a low carbon cold-worked austenite is similar to that formed from the annealed state.

In contrast to the Fe-Ni alloys, the addition of carbon greatly alters the morphology of martensite formed from cold-worked austenite. The yield strength of martensite formed from the cold-worked austenite of Fe-Ni-Cr-C alloys is greater than that of the martensite formed from the annealed austenite. The inheritance of the defect structure of the deformed austenite may result in several strengthening effects. Examples are: the increased nucleation sites for carbide precipitation during quenching and tempering; and the direct inheritance of dislocation networks in the martensite itself.

Kelly and Nutting suggest that the strength of martensite formed from cold-worked austenite is due to the lowering of the M_s temperature by plastic deformation resulting in a higher density of fine twins within the martensite. However, experimental determination of the M_s temperature by the use of the well known Greninger-Troiano technique clearly indicates no appreciable change in transformation temperature. The possibility remains, however, that the fine structure might form within the martensite as a consequence of the mechanical-thermal processing.

Change in the Tensile Properties of Zone Melted Iron With Decreasing Temperature: *G. J. London* and *Marvin Herman*, Franklin Institute Laboratories, and *J. L. Rutherford*, University of Pennsylvania.

Studies are in progress investigating the tensile behavior of purified iron prepared by the floating zone technique. Ten zone passes in high purity hydrogen through a 1 in. diam puron bar were used to produce the starting stock for the study. Of primary interest in the study is the variation in yield and strain hardening behavior with temperature from room temperature to −196°C. Preliminary data indicates that, although yield strength increases continuously with decreasing temperature, the strain hardening exponent appears to go through a broad minimum in the vicinity of −70°C.

This work was supported by the American Iron and Steel Institute.

Effect of Small Phosphorus Additions on the Recrystallization and Tensile Properties of Zone Melted Iron: *G. J. London*, Franklin Institute Laboratories.

Puron after ten floating zone passes in high purity hydrogen has been alloyed with phosphorus in the range zero to 0.06 wt pct. Recrystallization behavior has been studied from 600° to 750°C at various phosphorus levels. Tensile properties have been measured at 25°, −72°, −190°, and −268.8°C also at several selected phosphorus levels.

Preliminary results show that recrystallized grain size is refined, yield and tensile strength increased with increasing phosphorus content. Ductility generally decreases except in the vicinity of −70°C where tentative results indicate inverse dependence of ductility on phosphorus content.

This work was supported by the American Iron and Steel Institute and United States Air Force.

Microstructure of Fibrous Tungsten and Iron: *J. F. Peck* and *D. A. Thomas*, Massachusetts Institute of Technology.

Fibrous microstructures and their development have been studied by metallography and by hardness and quantitative metallographic measurements. Transverse sections of fibrous tungsten wire show thin, curved grain shapes rather than the expected equiaxed grains. The origin of this structure was investigated by drawing one iron rod to 87 pct reduction in area and swaging a second rod to the same reduction.

Specimens from the drawn rod at intermediate stages showed the development at large reductions of transverse-section microstructures similar to those in tungsten. The unusual grain shapes are attributed to the [110] texture of heavily worked bcc metals, which aligns <111> slip directions such that the grains can reduce in diameter in only one transverse direction; hence the grains become approximately elliptical in crossection. Swaged iron wire attains a *spiral nebula* structure that appears to result from the elliptical shape of the crossection of the swaging dies and their rotary motion.

Quantitative metallographic results parallel the changes in microstructure. The number of boundary intercepts per unit length is larger than expected, because of the grain shapes and also the appearance of deformation-band boundaries. Hardness measurements show an increased rate of work hardening above about 70 pct reduction, in agreement with some data in the literature. This probably results from the increased difficulty of further deforming a wire with a well-defined [110] texture, and it may be partly responsible for the very high strength of fine wires of bcc metals, notably tungsten and music wire.

This research was sponsored by the United States Air Force, Wright Air Development Division, Contract Number AF33(616)-6838.

Structure of Supercooled Nickel Ingots: *G. A. Colligan, V. A. Suprenant*, and *F. D. Lemkey*, United Aircraft Corp.

Electrolytic nickel has been inductively melted under an argon atmosphere in a variety of crystalline ceramic and fused silica crucibles. These melts have been supercooled prior to solidification over the range 27° to 290°C. Initial experiments with crystalline materials indicated that thoria, zirconia, and zircon crucibles permitted the nickel melts to supercool to a maximum of 156°C below the equilibrium melting point. In order to study ingots which had been supercooled to even lower temperatures, fused silica crucibles were used as outlined by J. L. Walker. The results of experiments using this technique indicate that the use of a noncrystalline container for the metal permits supercooling 200 to 300 g melts within 20°C of the empirical maximum that has been attained using micron size droplets.

The grain diameter as measured on the surface of the ingots exhibits a pronounced decrease between 145° and 162°C supercooling which confirms the earlier work of J. L. Walker. At less than 145°C, supercooling the coarse surface grain size appears to be more random than previously reported. The microstructures of these ingots have been studied in detail as well as the macrostructures.

For the experimental technique used in this investigation, superheat and time at superheat do not exert a strong influence on the amount of supercooling that is obtained prior to nucleation and solidification.

THURSDAY, OCTOBER 26

The audible pressure wave generated in these supercooled melts has been monitored using an acoustical technique. These measurements characterize the wave as approximately 200 cps with amplitudes as great as 180 db. No correlation of amplitude of this wave with supercooling has been found. The initial pulse of the wave corresponds to a collapse of the liquid metal surface.

Temperature Dependence of Tensile Properties of Binary Columbium Alloys: *G. D. Gemmell,* E. I. du Pont deNemours and Co.

The influence of temperature on the tensile properties of pure columbium and some binary alloys with titanium, zirconium, molybdenum, and tungsten from 70° to 2000°F have been determined. Strain aging effects are discussed.

True stress-true strain data from yield to maximum load and least squares values for m the strain hardening coefficient where

$\sigma = K\varepsilon_p^m$
σ = true stress
ε_p = true plastic strain
K = constant

were calculated by means of a computer program. The data for columbium are compared with published data for other refractory metals.

Tensile properties of Cb-Ti, Cb-Zr, Cb-Mo, and Cb-W alloys and effects of increasing solute on the yield strength of these alloys at various temperatures are shown. Titanium strengths only at temperatures up to 1200°F, whereas molybdenum, tungsten, and zirconium are effective to at least 2000°F.

Cb-10 at pct Zr and Cb-20 at pct Ti are ductile at room temperature. The addition of 10 at pct of either molybdenum or tungsten to columbium raises the ductile-brittle transition temperature to about room temperature.

Practically all alloys showed a region of hot shortness in forgeability at temperatures near 2000°F.

The significance of strain aging in columbium alloys was strongly apparent throughout this study. Discontinuous yielding toward the lower end of the temperature scale was observed in all systems. Ultimate tensile strength has shown inverse temperature dependence in the range of 400° to 1200°F in the pure metal and several alloys. Maxima in strain hardening with variation in temperature were observed. Distinct minima in strain rate resistivity n where

$$n = \left.\frac{\log \sigma_1}{\log \sigma_2}\middle/\frac{\log \dot{E}_1}{\log \dot{E}_2}\right| \quad E, T$$

were observed for several alloys. In this study, the two strain rates $\dot{E}_1$ and $\dot{E}_2$ were 26.7 and 0.067 min^{-1} respectively. Serrated load-elongation curves, as previously described for columbium and other strain aging metals, have been observed throughout this study.

Mechanical Properties of Cb–Zr–W and Cb–Zr–Mo Alloys: *A. G. Scott* and *W. I. Pollock,* E. I. du Pont de Nemours and Co.

This paper is concerned with the high temperature strength properties of columbium alloys containing zirconium, molybdenum, and tungsten. Tensile and creep rupture properties have been determined for compositions containing up to 15 wt pct total alloy addition. In addition, TIG weld ductility has been examined along with elevated temperature tensile strength of welded specimens.

Effects of interstitial content on fabrication and elevated temperature strength are discussed. The effect of carbide dispersions is shown. Further, the temperature of mechanical processing has been found to be an important variable.

Two columbium alloy compositions have been extensively evaluated for sheet and tubing applications. The alloys are characterized by tensile and rupture strengths greater than Cb-1Zr, excellent fabricability, and good weld efficiency.

Influence of Rolling Temperature on the Cryogenic Properties of Type 300 Series Stainless Steel: *E. B. Mikus,* General Dynamics Corp.

The affect of elevated temperature rolling and subzero temperature rolling on the tensile and toughness properties of 301 and 302 stainless steel at cryogenic temperatures was determined. Rolling temperature varied from −320° to +400°F, and the degree of reduction ranged from 5 to 75 pct. The toughness resulting from these processes was evaluated using a criterion of notched tensile to unnotched tensile strength at room temperature, −320° and −423°F.

The degree of transformation obtained by the various rolling procedures was determined by magnetic measurements using a calibrated Magna-Gage. Electron micrographs were obtained on the various microstructures to corroborate the degree of austenitic transformation and to ascertain the mode and type of martensite formed by the various rolling procedures.

The results indicate that superior toughness at cryogenic temperatures may be obtained by rolling 301 SS above the M_d temperature prior to room temperature rolling compared to commercially rolled material which is rolled completely below the M_d temperature.

Subzero rolling 301 SS on the otherhand produced a structure that was very brittle at −423°F.

With 302 SS excellent strength and toughness was obtained at −320°F on material subzero rolled at −100°F as well as −320°F.

The electron micrographs of structures produced by subzero rolling suggest a different mode of martensite distributions than that produced by rolling at elevated temperatures. However, the interpretation of this different structural distribution to account for the lower toughness observed is not clear. The toughness data obtained on 301 SS at cryogenic temperatures suggests that an optimum amount of martensite is necessary to assure good toughness at high strength levels.

A Study of the Effects of Nuclear Radiation on High-Strength Aerospace Vehicle Materials at the Boiling Point of Hydrogen (−423°F): *J. F. Watson, J. L. Christian,* and *J. W. Allen,* General Dynamics Corp.

The problem of radiation damage occurring in structural materials at −423°F became of major importance during the studies of nuclear powered upper stage rockets. In this type of vehicle, large propellant tanks containing liquid hydrogen at −423°F are subjected to large doses of both neutron and gamma fluxes. Thus the structural materials experience a combined environment of cryogenic temperature and nuclear radiation. To study the nature of these effects, samples were exposed to a fast neutron flux of about 2×10^{17} ($E > 0.33$ mev) nvt while being soaked in liquid hydrogen (−423°F). After the radiation exposure the samples were subjected to tensile tests while being held at −423°F without intervening warmup. The alloys studied were 301 and 310 stainless steels cold-rolled 60 and 75 pct respectively, 5Al-2½ Sn titanium (AllOAT), and 2014-T6 aluminum. The tensile tests performed gave tensile strength, elongation, notched tensile strength ($K_t = 6.3$), and the tensile strength of simple heliarc butt welded joints. Samples were tested in the unirradiated and radiated conditions at both 70° and −423°F.

The largest and most consistent effect was a decrease in tensile strength after irradiation at −423°F. This effect varied from a 27 pct decrease for the 301 stainless steel to a 3 pct decrease for the 5Al-2½ Sn titanium. It appears that the effect of cryogenic irradiation is to severely restrict the amount of plastic deformation the samples can sustain after yield and prior to fracture. This phenomenon would be in accordance with the theory that neutron radiation acts to introduce vacancies and interstitials which restrict the amount of plastic strain the samples can withstand before fracture. This effect is believed to be more severe at cryogenic temperatures because the neutron generated vacancies and interstitials can diffuse out at the higher temperatures, but are *frozen* in at cryogenic temperatures. Notched tensile tests showed that small amounts of embrittlement occurred in the 301 stainless steel after cryogenic irradiation, while the 310 stainless steel showed no embrittlement. It is believed that this condition resulted from a radiation induced solid state phase transformation of austenite to the more brittle martensite phase in the 301 stainless steel. No such transformation occurs in Type 310 stainless steel. The titanium and aluminum alloys showed no embrittlement as a result of the cryogenic irradiation.

Viscosities of Liquid Bismuth Lead and Zinc to 1000°C: *Donald Ofte* and *L. J. Wittenberg,* Monsanto Research Corp.

The viscosities and activation energies for viscous flow of bismuth, lead, and zinc to 1000°C are reported as measured in an oscillating cup viscosimeter. The absolute viscosity of the metal at a particular temperature was calculated from the logarithmic decrement and the period of the oscillating torsion pendulum was determined by a photo-electrically operated clock to one-thousandth of a second. The amplitudes of the oscillations, which were used to calculate the logarithmic decrement, were measured by a photographic technique with a precision of one part in ten-thousand. The metal under investigation was sealed in the oscillating cup so that vaporization loss and oxidation of the liquid metal was prevented. No interactions were noted between the liquid metals and the tantalum cup.

The logarithms of the viscosities of bismuth, lead, and zinc exhibit a first-order dependency as a function of reciprocal temperatures. The equations of the lines generated from this type of plot are:

Bismuth	$\log_{10} \eta = 0.3368\ (1/T) - 0.3510$
Lead	$\log_{10} \eta = 0.4506\ (1/T) - 0.3338$
Zinc	$\log_{10} \eta = 0.6625\ (1/T) - 0.3841$

where η = viscosity (centipoise) and $T = {}^\circ K \times 10^{-3}$

The viscosities were measured to within 1° of the melting point of zinc, 2° of the melting point of lead and in the supercooled condition, of bismuth, 9° below the melting point.

The activation energy for viscous flow decreases from zinc to lead to bismuth and is constant for each of the liquid metals over the measured temperature intervals. Since the activation energy for viscous flow constitutes a measure of the order in the liquid, no abrupt change is observed in the ordering of liquid bismuth, lead, and zinc when their melting points are approached, as contrasted to the reports of some previous investigators. For bismuth the same type of order present in the thermodynamically stable liquid persists into the supercooled state.

Powder Metallurgy and Processing of Metals

2:00 to 5:00 pm **Sky Room**

(Based on Submission of Research Abstracts)

Chairmen:

Marion Semchyshen, Climax Molybdenum Co., and *J. Wulff*, Massachusetts Institute of Technology.

Shrinkage Behavior of Copper Powder Compacts: *F. V. Lenel, H. H. Hausner, G. S. Ansell, I. A. ElShanshoury*, and *J. G. Early*, Rensselaer Polytechnic Institute.

The shrinkage behavior, during the initial stages of sintering, of a group of pressed irregular cooper powder compacts was determined by a dilatometer method.

The effects of pressing pressure, sintering temperature, and external load during sintering upon shrinkage were determined. In addition, determination was made of the residual stress present on the surface of the compacts during shrinkage by X-ray techniques. In particular the following observations were made: 1) Sintering of the compacts begins at a much lower temperature than would be expected; 2) Residual stresses arising during powder compaction remain even after shrinkage occurs; 3) External loading has a marked effect both on the temperature at which shrinkage occurs and the absolute value of shrinkage obtained; and 4) The rate controlling mechanism for sintering changes with sintering temperature.

These results are discussed in terms of the theoretical models which have been proposed to account for sintering. It appears that none of these models adequately explains the observed results. A deformation model for shrinkage is proposed which qualitatively accounts for the observed results.

This work was supported under a grant from the Atomic Energy Commission.

Sintering of Nickel-Base Superalloys: *E. J. Westerman*, General Electric Co.

Prealloyed powders of nickel-base superalloys, including the René 41, Udimet 500, and Nimonic 100 compositions, were sintered to almost theoretical density in short sintering times. It was ascertained that the rapidity of densification was allowed by the presence of a small amount of liquid phase at the grain boundaries, apparently originating by incipient melting in these regions. It was experimentally determined that the liquid phase formed in each alloy at temperatures considerably below the temperatures at which macroscopic melting of the compacts occurred, allowing rapid liquid-phase sintering without gross deformation of the compacts.

The dependence of room-temperature tensile properties on sintering temperature and time was determined for the René 41 and Udimet 500 alloys. Although the 0.2 pct offset yield strength of the Udimet 500 alloy was superior to that of the corresponding wrought alloy, all other properties of the sintered alloys were inferior. All ductilities were low. An increase in the quantity of liquid phase present during sintering, caused by an increase in sintering temperature, increased the densification rate; the room temperature tensile properties, however, were thereby decreased. Optimum properties were obtained by sintering for long times with relatively small amounts of liquid phase present.

Particulate Joining: *P. D. Johnson*, The Glidden Co.

Brazing and soldering are normally identified with solid shape copper and silver solders and lead-tin alloys, respectively, functioning as the joining medias.

Powders, usually in paste form, are proving increasingly popular. Examples are cited in the furnace, torch, induction and dip joining of ferrous metals, including stainless, and brass, copper and aluminum. Advantages are detailed, especially as they relate to methods of application, built-in flux control and unique adaptability to specific applications.

Brazing and soldering pastes always consist of a joining media, a vehicle and very possibly a flux depending upon the nature of the filler and parent metals and the techniques to be employed. Careful consideration is given to the inter-relationship of these variables as they bear specifically upon formulations.

Expanded use of powdered metals and oxides will come about through continuing improvement of existing materials and relentless search for theoretical knowledge of the problems at hand. A look into the future is attempted with a statement of some of the basic problems and possible solution approaches. Although of necessity this involves broad spectrum consideration, emphasis is placed on powders and their potentiality in the bonding of a variety of metals. A closer view is taken of the functions of a flux, including chemical and physical requirements, and the compatibility essentials of particular parent and filler metals perceived in terms of the solid state.

Comments will be augmented with slides offering tabulated data and illustrating a variety of particular joining operations.

Vacuum Arc Skull Casting of Tungsten-Molybdenum Alloys and Pure Tungsten: *A. E. La Marche*, Westinghouse Electric Corp.

The vacuum-arc skull casting of pure tungsten and tungsten-molybdenum alloys using dc straight polarity as the source of power is described. The paper outlines the data accumulated in evaluating the vacuum-arc skull casting technique as a method of producing tungsten and tungsten alloys for rocket nozzles. This work was conducted on US Navy Bureau of Naval Weapons Special Projects Office, Contract No. NORD-18791-(FBM). Three in. diam bars of the 50 tungsten-50 molybdenum, 70 tungsten-30 molybdenum, 90 tungsten-10 molybdenum wt pct alloys, and pure tungsten in the pressed and sintered condition were vacuum-arc melted and skull cast into special high purity machined graphite molds designed and produced to mold the fluid tungsten. Destructive testing of the resultant parts was conducted to determine hardness, soundness, contamination, and grain size of the castings.

The first pure tungsten skull castings ever produced have been made by Westinghouse in conducting this development program for the Navy. The ability to vacuum arc melt and cast 60 to 70 lb pure tungsten castings was demonstrated.

The reclamation of tungsten scrap by remelting was achieved with very promising results. Attempts to reclaim tungsten-molybdenum alloy scrap however met with little success. This lack of success stemmed from the fact that a non-consumable electrode has not been found to accomplish the pigging or purification of this material.

The temperature of the tungsten arc has been calculated theoretically and confirmed in experimental melting to be in the order of 8000° to 11,000°F. In addition the partition of power or the amount of power which is received by the cathode and the anode has been shown to be approximately 10 to 25 times greater at the anode or bath than that received by the cathode or consumable electrode using dc straight polarity.

Analytical techniques have been used which allow a good chemical analysis of the pure tungsten to be made prior to and after melting so the purity associated with the pressed and sintered, the vacuum-arc skull casting, and electron beam zone refining using both metallographic and chemical techniques is briefly discussed.

Molybdenum-0.5 pct. Titanium Grain Refinement: *D. R. Carnahan, G. Reimann*, and *S. Inouye*, Westinghouse Electric Corp.

A consumable vacuum arc furnace capable of melting up to 6 in. diam ingots was installed at WADD, Wright-Patterson Air Force Base, Ohio. This furnace was designed to enable it to melt refractory metals without furnace failure due to poor water cooling, voltage, insensitivity, and arc instability.

A total of 30 heats have been cast in this furnace to date. The first three heats being plain carbon steel. These first three heats were cast to check out the furnace's vacuum and electro-mechanical capabilities before any refractory metals were melted in an untried furnace. Twenty-five, Mo-0.5 pct Ti heats were cast in the furnace to study titanium homogeniety and the effect of

THURSDAY, OCTOBER 26

magnetic stirring and arc pulsation on as-cast grain size. Two heats of Mo-0.5 pct Ti were cast at Materials Manufacturing dept., Westinghouse, Blairsville to study the effects of ultrasonics on as-cast grain size.

Electrode material used in this work was 2½, 2, and 1¾ in. diam by approximately 20 in. in length with a ⅛ in. hole through the center axis of the electrode. The composite electrode was fabricated simply by placing ⅛ in. diam titanium rods down through the center of the hollow molybdenum electrode.

In all heats that were stirred, grain refinement resulted regardless of type of stirring.

In ingots that were treated ultrasonically, grain refinement resulted but the control of ultrasonic equipment was very difficult to maintain as pertaining to sensitivity which resulted in inconsistent as-cast grain structures. Arc pulsation resulted in little, if any, grain refinement.

Molybdenum Forging Process Development: *John Bonchak,* Westinghouse Electric Corp.

One of the most serious difficulties currently experienced by the forging industry is to produce molybdenum alloy forgings having isotropic elevated temperature properties which would meet the engineering needs of today and the near future.

An economically feasible (favorable material yields of 83 pct) technique has been developed for the direct hot-work forging of arc-cast TZM alloy ingots. The process consists of heating the material to its relatively plastic range (1900° to 1950°C) in an argon atmosphere and forging in air.

Because this alloy is responsive to heat treatment, it has been found that proper primary working temperatures are conducive to outstanding elevated temperature strengths in the as hot-worked condition. A given amount of secondary working at about 1400°C improves the room temperature tensile strengths with little influence on the high temperature strength properties.

In the process of producing hot-forged billets the following observations were made:

1) a resonably good as-cast ingot need not be surface conditioned prior to forging;
2) forging in air at hot-work temperatures does not cause surface tears nor serious atmosphere contamination of the work piece; and
3) grain size in itself could not be considered a criterion for passing judgment on the relative strength of this alloy.

Of significant importance in improving the strength properties is the size structure and distribution of the carbide phase.

Among the outstanding strength values at 2400°F displayed by the alloy after the hot-work process are:

1) 84,100 Ult TS-15.1 pct Elongation; and
2) 100 hr rupture lift at 30,000 psi stress level.

The process was developed under sponsorship of AMC, Aeronautical Systems Center, Wright-Patterson Air Force Base.

The Planetary Mill as a Research Tool: *C. L. Kolbe,* General Electric Corp.

The Sendzimir hot planetary mill is a new concept in hot rolling thick slab ingots directly to strip in one rolling operation. The unique capability of producing reductions greater than 90 pct is obtained by pushing the slab ingot into the roll bite and taking many small reductions with multiple work rolls rotating at high speed. Slab ingots 3 to ¾ in. thick can be reduced to strip less than ⅛ in. thick in a single furnace heating, depending on the particular mill design. Two series of work rolls, supported in synchronized cages, are rotated around back-up support and drive rolls. The cages rotate so that approximately 70 pairs of rolls a second pass through the roll bite driving forward small increments of material with each work roll.

The Sendzimir planetary mill is popular in Europe and Canada as a substitute for continuous tandem ferrous strip mills. In the United States, three smaller planetary mills have been placed in operation, and two of these are in research laboratories. One of these is located at the General Electric Research Laboratory.

A series of experiments have been performed to study the planetary mill as a research tool in the rolling of ferrous, nickel base and refractory metals. The advantages of single heating, contamination, low temperature loss, low rolling loads, and compression rolling were balanced against sample size limitations and complexity of mill operation in producing small amounts of sample sheet material.

The mechanical properties of several materials were compared, and these properties were studied with the ease or difficulty of rolling on the planetary mill in view. The variations in rolling load were recorded and examined in order to understand better the operation of the planetary mill.

Commercial materials of all types can be processed on a planetary mill. Refractory materials and special alloys which tend toward segregation and casting defects are more difficult to roll and require special tooling and heating techniques.

Recognition of NATIONAL SCIENCE FOUNDATION Grant

Participation by foreign speakers in the symposia on "High Purity Iron and its Dilute Solid Solutions" and "Effects of Surface and Environment on Strength," held on Monday and Tuesday, October 23 and 24 was made possible through the financial support extended by the National Science Foundation. The Metallurgical Society of AIME wishes to acknowledge, and express its appreciation for, the continuing support of its activities by the Foundation.

INDEX OF AUTHORS AND CHAIRMEN

A

Aaronson, H. I., 23
Achter, M. R., 13, 28
Adams, M. A., 14
Alden, T. H., 19
Allen, B. C., 14
Allen, J. W., 40
Allen, N. P., 12
Allen, Steven, 14
Ansell, G. S., 10, 19, 41
Armstrong, R. W., 10, 26
Arnold, E. D., 22
Arora, O. P., 21
Arrott, Anthony, 8
Ashoff, W. A., 15
Avery, D. H., 20

B

Backofen, W. A., 20
Badiali, M., 8
Bakish, R., 8
Baluffi, R., 23
Banks, C. N., 20
Bardos, D. I., 24
Baroch, E. F., 15
Barr, R. Q., 17
Barrett, C. S., 11
Basinski, Z. S., 7, 20
Bechtold, J. H., 14
Beck, P. A., 24
Belk, J. A., 16
Bendler, H. M., 29
Berghezan, A., 11
Birchenall, C. E., 21
Birnbaum, H. K., 9
Boedtker, O. A., 32
Bolling, G. F., 26, 35, 36
Bonchak, John, 42
Brand, G. E., 21
Bratina, W. J., 39
Brigham, R. J., 23
Brittain, J. O., 26, 38
Brophy, J. H., 9
Brown, Norman, 9, 26
Burr, A. A., 31
Burris, Leslie, Jr., 18, 22
Bush, R. H., 29
Butler, J. F., 29
Butler, T. E., 15

C

Cabrera, N., 7
Cairns, R. E., Jr., 17
Campbell, R. A., 20
Carlson, R. G., 8
Carnahan, D. R., 41
Chadwick, G. A., 36
Chalmers, Bruce, 33
Chang, W. H., 17
Chao, Pao Jen, 6
Chaudhuri, A. R., 28
Chipman, John, 25
Choi, Jei Y., 32
Christensen, E. L., 22
Christian, J. L., 40
Clark, J. B., 37
Clark, J. W., 16
Clark, Robert, 12
Clark, W. E., 23
Cline, R.S., 34
Cohen, J. B., 37
Colligan, G. A., 39
Conrad, Hans, 27
Cost, J. R., 25
Cotner, J., 9
Coyne, J. E., Jr., 31

D

Dancy, T. E., 15
Darby, J B., Jr., 21, 24, 25
Das, B. N., 24
Das Gupta, S. C., 38
Dash, John, 31
Davis, R. S., 8, 30, 33
Dean, O. C., 22
DeKany, J., 22
Doherty, P. E., 33
Donaldson, D. M., 22
Dove, D. B., 20
Downey, J. W., 24
Dresher, W. H., 15
Drew, J. B., 32
Dunn, C. G., 34
Duwez, Pol, 32, 36

E

Early, J. G., 41
Elbaum, C., 18
Ellis, W. C., 33
ElShanshoury, I. A., 41
Emerick, H. B., 15
Endebrock, R. W., 16
Enrietto, J. F., 29
Ernst, W. S., Jr., 22

F

Fenn, E., 8
Ferguson, D. E., 22
Ferris, D. P., 14, 27
Fine, M. E., 37
Fleischer, R. L., 9
Floreen, S., 13
Foldes, Stephen, 6
Fopiano, P. J., 38
Foster, E. L., 16
Fountain, R. W., 25
France, L. L., 8
Frank, R. G., 8
Franklin, Wilbur, 10

G

Gemmell, G. D., 38, 40
Gensamer, Maxwell, 12
George, F. D., 37
Giannakos, F. P., 8
Gilman, J. J., 7, 13
Gjostein, N. A., 32
Gokcen, N. A., 20
Goodrich, R. S., 10
Grace, R. E., 17
Grant, N. J., 15, 17
Gregory, D. P., 18, 19
Greiner, E. S., 33
Grube, W. L., 39
Gulbransen, E. A., 25
Gullett, W. W., 15
Gunow, R. F., 20
Gupta, K. P., 24
Guthrie, C. E., 18
Gutowski, J. A., 33

H

Haak, Fritz, 25
Hablanian, M. H., 20
Halliday, I. M. D., 12
Hartley, K., 22
Hausner, H. H., 41
Haverstraw, Robert, 32
Hayden, H. W., 9
Head, A. K., 11
Hendrickson, I. G., 30
Herman, Herbert, 37
Herman, Marvin, 39
Hiltz, R. H., 21
Hirsch, H. H., 6
Hirth, J. P. 7, 24, 32
Holden, F. C., 14
Holland, J. R., 35
Holt, D. B., 11
Hornbogen, E., 38
Hornell, C. A., 16
Hu, Hsun, 34, 35
Hucke, E. E., 13
Huffstutler, M. C., Jr., 25, 32
Huggins, R A., 29
Hultgren, R. R., 25

I

Imgram, A. G., 14
Inouye, S., 41

J

Jaffee, R. I., 14
Jahnke, L. P., 17
James, D. B., 22
Johnson, K. W. R., 18
Johnson, P. C., 30
Johnson, P. D., 41
Johnston, T. L., 9, 27, 28
Johnston, W. C., 35
Jones, E. S., 6
Jones, R. G., 20
Jones, R. L., 30
Jugle, D. B., 21
Justusson, W. M., 39

K

Katz, O. M., 25
Kauffmann, J .W., 37
Kaufman, Larry, 30
Keh, A. S., 38
Kellogg, H. H., 12
King, A. H., 11
King, H. W., 24
King, T. B., 15
Kirkaldy, J. S., 23
Klement, W., Jr., 36
Klodt, D. T., 17
Klopp, W. D., 6
Kolbe, C. L., 42
Koo, R. C., 26
Kraft, R. W., 36, 37
Kramer, I. R., 11
Kramer, J. J., 35, 36
Krivsky, W. A., 20
Kronberg, M. L., 9
Ku, R. C., 26
Kula, E. B., 34
Kulin, S. A., 38

L

Lally, F. J., 21
Lam, D. J., 24, 25, 31
La Marche, A. E., 41
Lautenschlager, E., 38
Lavendel, H., 22
Lawley, A., 14
Lawson, Walter, 15
Leary, J. A., 18, 21
Lees, P., 22
Lement, B. S., 14
Lemkey, F. D., 37, 39
Lenel, F. V., 41
Leslie, W. C., 12
Levinstein, M. A., 6
Li, C. H., 27, 28
Li, J. C. M., 20, 35
Liu, Y. C., 34
Livingston, J. D., 10
London, G. J., 39
Lopata, S. L., 34
Lothe, J., 7
Low, J. R., Jr., 12
Lowney, J. W., 25
Lundy, T. S., 24
Lusby, W. E., Jr., 15

M

Ma, C. H., 23
Machlin, E. S., 7
Maddin, R., 14
Maltz, Joseph, 14
Massalski, T. B., 24
Maykuth, D. J., 14
McCabe, C. L., 8, 12
McCoy, H. E., 30
McEvily, A., 28
McLaren, E. H., 36
McNeish, W. A., 8
McNutt, J. E., 38
McPherson, D. J., 12
Metzger, M., 21
Michalak, J. T., 12
Mikus, E. B., 40
Mills, D., 29
Minkler, W. W., 12, 15
Moran, Dana, 15
Morgan, R. P., 15
Mueller, C. P., 8
Murdock, J. F., 24
Murray, G. T., 13

N

Nachman, J. F., 17
Nakayama, Y., 14
Nanis, L., 11
Necker, W. C., 20
Nemethy, L., 12
Nevitt, M. V., 24
Nichols, H., 8
Nielsen, J. P., 33
Noakes, J. E., 8
Norton, L. J., 25

O

Ofte, Donald, 40
Ogden, H. R., 6, 14, 16
Orehotsky, J. L., 16
Otte, H. M., 31, 37

P

Park, B., 8
Parkinson, N., 22
Passmore, E. M., 14
Patel, J. R., 28
Pearson, J., 12
Peck, J. F., 39
Peiffer, H. R., 28
Perlmutter, I., 8
Pettit, F. S., 21
Pfann, W. G., 35
Pierce, R. D., 18
Pollock, W. I., 40
Pound, G. M., 7
Priest, D. K., 6
Pulsifer, Verne, 8
Purdy, G. R., 23
Pye, J. J., 32

R

Radcliffe, S. V., 31
Ragone, D. V., 13, 21
Ramagopal, G. R., 21
Rapp, R. A., 25, 33
Raymond, P. L., 16
Reavis, J. G., 16
Reimann, G. R., 41
Rhines, F. N., 7
Richman, R. H., 33
Rieppel, P. J., 15
Robertshaw, T. L., 8
Robertson, W. D., 32, 38
Roblin, M. J., 10
Rogers, R. R., 20
Rometsch, R., 22
Rose, R. M., 14, 27
Rosenbaum, H. S., 9
Rostoker, W. H., 8
Rowe, G .H., 18, 19
Rutherford, J. L., 39
Rutter, J. W., 33

S

Salkovitz, E. I., 16
Savage, J., 12
Sayre, E. D., 6
Schatz, M., 31
Schmatz, D. J., 39
Schoenfeld, W. J., 8
Schrader, C. F., 15
Schulman, J. H., 11
Schultz, J. M., 10
Scott, A. G., 40
Seltzer, Martin, 23
Semchyshen, Marion, 17, 41
Shewmon, P. G., 23, 32
Shyne, J. C., 39
Silcox, John, 7
Simons, J. C., Jr., 16
Smith, R. L., 8
Snowden, K. U., 29
Speich, G., 30
Spretnak, J. W., 30

INDEX OF AUTHORS AND CHAIRMEN

Srp, O. O., 6
Stein, B. A., 30
Stevens, J. I., 18
Stokes, R. J., 13, 27, 28
Stone, L. P., 33
Suprenant, V. A., 39
Swalin, R. A., 23

T

Tanner, L. E., 31
Tetelman, A. S., 38
Thomas, D. A., 39
Thomas, Gareth, 32, 37
Tiller, W. A., 35, 36
Torti, M. L., 16
Treuting, R. G., 33
Tufts, C. F., 17

U

Underwood, E. E., 27

V

Van den Sype, J., 14
van der Merwe, J. H., 7
Van Westenberg, J. A., 8
von Destinon-Forstmann, J., 23

W

Wagner, J. B., Jr., 10, 21, 23
Wagner, R. S., 33
Walling, M T., 18
Walter, J. L., 34
Warlimont, H., 31
Waterstrat, R. M., 24
Watson, J. F., 40
Weart, H. W., 35
Weertman, J., 9
Weinberg, F., 36
Weir, J. R., 30
Weissmann, S., 14
Wells, Cyril, 15
Wells, M. G. H., 32
Wert, C. A., 25
West, D. R. F., 32
Westerman, E. J., 41
Westbrook, J. H., 7
Westwood, A. R. C., 13
Wilkinson, W. D., 17
Willens, R. H., 31, 36
Wilsdorf, H., 37
Winterbottom, W. L., 24
Wittenberg, L. J., 40
Wood, W. A., 29
Wriedt, H. A., 38
Wulff, J., 9, 14, 27, 41

Y

Young, W. R., 6
Yue, A. S., 37

Z

Zackay, V. F., 39

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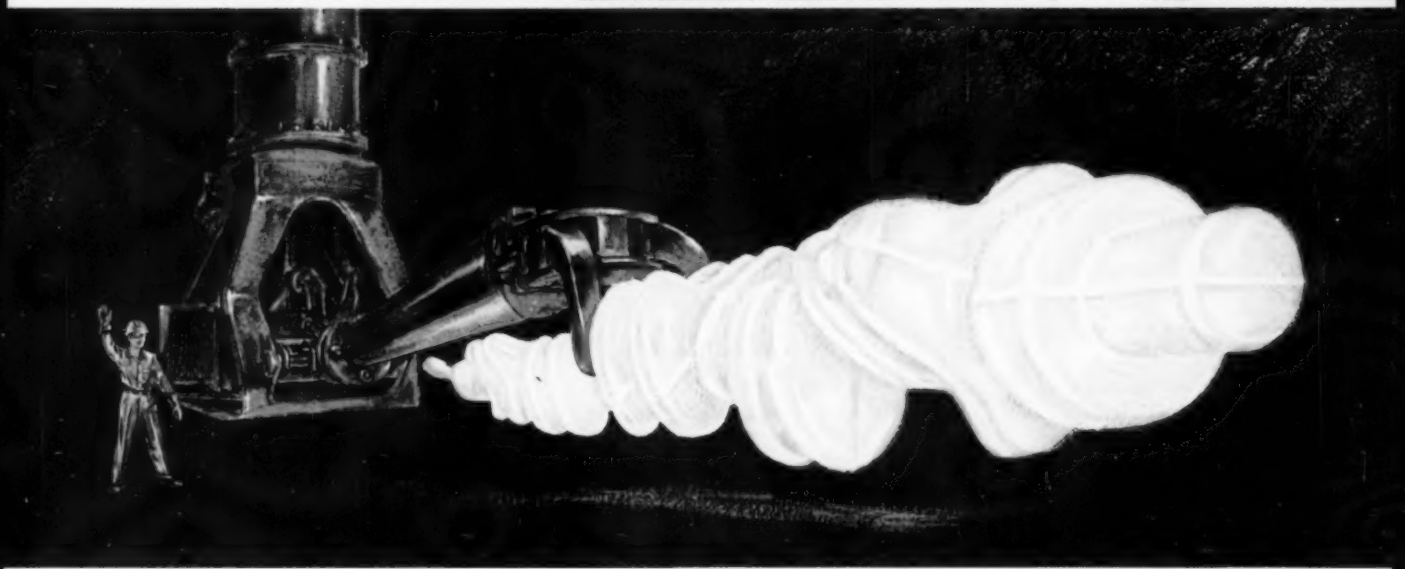

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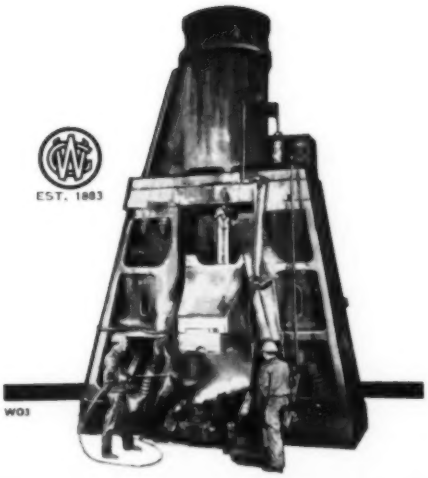

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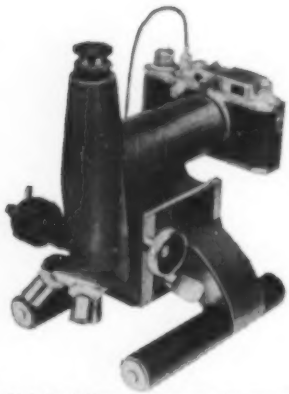

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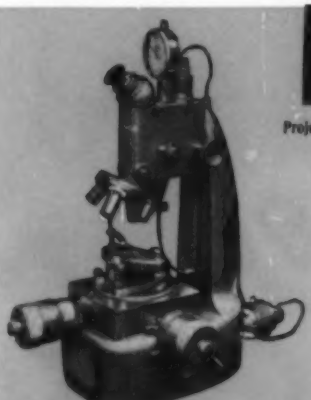

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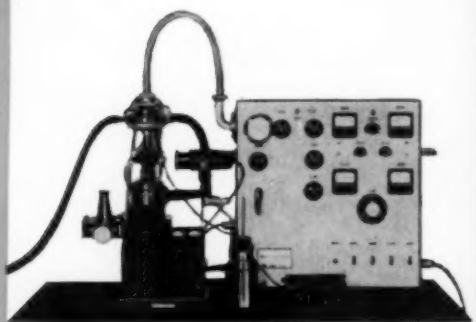

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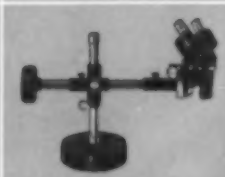

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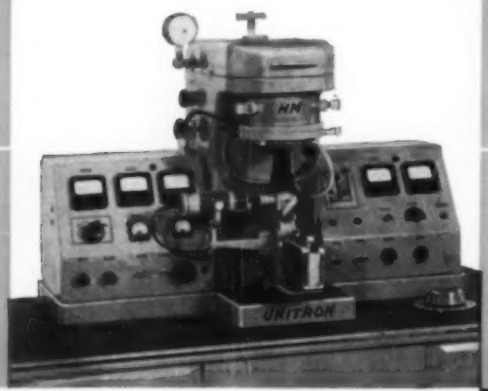

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